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LECTURES  
ON  
PRACTICAL PHARMACY



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LECTURES  
ON  
PRACTICAL PHARMACY

BY  
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NEWCASTLE-UPON-TYNE



LONDON  
J. & A. CHURCHILL, NEW BURLINGTON STREET

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1873

RS 91  
873P

## PREFATORY LETTER.

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TO MY READERS,—A chapter of genesis, to explain why I have written and why I have published the following pages, will do something to justify their production in their present imperfect condition.

About a dozen years ago, when a threatening cloud passed over the pharmaceutical horizon, my neighbours met together for mutual protection; and the pharmaceutical assistants of the town took the opportunity of petitioning their elders for a course of local lectures on pharmaceutical subjects; in response to which a local school was instituted, Mr Brady accepting a lectureship in Botany, and I Pharmacy, for the first session; subsequent sessions being devoted to Chemistry and Materia Medica, which were expounded by other friends. The attendance, at first satisfactory, gradually diminished, till during the third year of the school's existence, I had the honour of reading a paper on sulphur, and performing the experiments in illustration, before an audience of *one*. Perseverance could no further go, and the local pharmaceutical school was relinquished; it was born before its time, and died in due course.

When the Pharmacy Act of 1868 came into operation, a general agitation of the education question resulted in the

establishment of local organisations all over the country. Bearing in mind our previous experience in Newcastle, some of us were rather reluctant to originate another institution, in the permanence of which we could not feel confidence, but as some who were interested in the subject thought that Newcastle should not be behind other towns in this good work, it became my duty as local secretary to call the members of our trade together with the view of discussing the subject. As the result of our meetings, the conclusion was arrived at, that thoroughness and permanence in the attainment of the desired object would be best secured by arranging, if possible, with the College of Medicine, to have the wants of the pharmaceutical student provided for, in conjunction with what was already supplied to medical students; to do which, only required that a course of lectures on Pharmacy should be added to the curriculum, which already included Botany, Chemistry, and *Materia Medica*.

The College of Medicine having readily agreed to give the proposed experiment a trial, and my fellow-pharmacists having pointed to me as the probable lecturer on Pharmacy, I was in due form elected to the office.

The post was one which I did not seek to obtain, nor feel at liberty to decline. It was my duty to do what I could for the general good, and as the department of Pharmacy was allotted me, it remained only for me to do my best to fill it to the satisfaction of the students who might hear me, and to the credit of the College which had been first to institute a lectureship in pure Pharmacy.

Prior to my becoming lecturer, I had never had the privilege of listening to any lectures on Pharmacy, *Materia Medica*, or Botany; I was therefore without a model, and

felt myself left pretty much to my own devices as to the selection of matter and the mode of its treatment. This was in *some* respects an advantage, as it left me more at liberty to include whatever I thought desirable—whatever I thought was required to complete the pharmaceutical curriculum, and was not already treated under the heads of Botany, Chemistry, or Materia Medica.

The aim most constantly and most prominently before my mind, was to educate, rather than to instruct my class—to expand and develope their ideas of the nature of our calling, and give vitality to its scientific character, rather than to store their minds with an accumulation of facts, cut, dried, and classified in natural orders like a herbarium.

How far I succeeded in providing acceptable matter it is very difficult for me to judge. On the one hand; I had many indications of the interest taken in the lessons by the students, and many inquiries for a good text-book on my subject. On the other hand, there was the unfavourable evidence that the number of students diminished from year to year. This latter circumstance soon showed that the lectures, be they good or bad, could not be regarded as a permanent institution, and indicated that if my efforts were to produce results commensurate with the labour they cost, it would have to be through seeking the wider field which publication would open out; and finding a score of probable readers here, I speculated upon the kingdom providing me with a thousand or more, if I could put my raw material into a more digestible condition. I was not blind to its many faults, nor to the difficulty I should have in making it ship-shape, and postponed the task from year to year, hoping for a more convenient season; but between a feeble constitution and an active business, the opportunity appeared

never to approach, and I at last resolved to do roughly and hastily, what I had not the leisure to do carefully and well; the choice appeared to be between a roughly executed task and none at all.

I have retained throughout the feeling that I am by nature a student rather than a teacher, and this fact will be found to have left its mark upon my work. The professed teacher will adopt a system, whether it be of nomenclature, notation, or any other appliance, and will teach throughout upon that system; while the habit of the professed student, more especially the self-taught student, absorbing educational aliment wherever it may be presented, and assimilating the mode of thought and expression of each authority in turn, will communicate his information in a manner which is characterised by variety, or even inconsistency, rather than uniformity. I have been led in this way to mix different thermometrical scales, and different weights and measures, as well as different nomenclatures and equivalent numbers, but I hope I have done so without involving any obscurity, or without requiring of the student a knowledge of any systems which are not necessarily included among his familiar possessions. And while I anticipate that some readers would prefer to have a uniformity of expression, I have satisfaction in knowing that I am not without the countenance of good authority in my practice.

In the *Pharmaceutical Journal* for March 22, 1873, the reviewer of "*Valentin's Analysis*" says:—"We have the same kind of objection, and one equally decided, to the invariable employment of systematic names, and indeed to the use of one class of names to the exclusion of others. As is very justly observed by Whately, the disposition to do 'this is that' which chiefly constitutes what is called narrowness of mind'



—a complaint from which chemists are no more exempt than other mortals.”

In all probability many of my readers, like myself, will be almost unconscious of the mixture of Latin and English, which trade customs have rendered so common that they cease to offend our ears, even though in theory we prefer to use our mother tongue without adulteration. I am conscious that every lecture would be better for further revision, and every subject treated of would bear extension ; many subjects about which there is difference of opinion, such, for example, as the phenomena of percolation, might have been treated more exhaustively ; but while I have endeavoured, in all such cases, to rely mainly upon my own observations, courses of experiments for the verification or correction of various subjects have been commenced at different times, when the pressure of other work was not excessive, but have not so often as could be wished terminated in conclusive results. The pursuit of daily bread is scarcely conducive to philosophical accuracy, and many experiments have been cut short by the interruptions of business, and the natural limit placed upon my work by a not over robust constitution.

Many of the official formulæ are passed over almost without comment, because of the remarks which I could make and qualify when addressing my class, being scarcely ripe for the reception of that greater importance which is too apt to be attached to printed matter ; but I have retained them, meagre as they are, with the idea that the lectures may be advantageously read aloud by the leader of any mutual improvement class, and supplemented by his own comments ; with the same view I have in many cases described the experiments with which I have endeavoured to illustrate the principles enunciated.

In treating of official products, I have commonly assumed that the commercial articles are obtained by the official formulæ, though it is well known that manufacturers do not usually follow the pharmacopœial instructions in the preparation of trade chemicals, it may be not always with identical results. A manufacturer may assume that granular sulphate of iron is simply protosulphate in small crystals, and consequently it is open to him to produce the same without the use of spirit, and supply it as "Ferri. Sulph. Gran. B. P.;" and without considering whether or not the products are absolutely identical, he is satisfied that it is equally good for pharmaceutical use.

Many of the lectures are much shorter than enough to occupy the usual hour in reading; this is the natural consequence of the time spent in illustrating with experiments, and I think it will scarcely be found that any one contains less matter than it is desirable to bring before a student at one lesson.

In preparing for the press I have preserved much of the colloquial style, which was natural in the lecture-room. I feel that I am still addressing friends and fellow-students, and with a feeling that a friendly greeting awaits the appearance of my work, I lay it before you with the conviction that whatever may be its shortcomings, it cannot fail to strengthen the bond which already links me to all students in Pharmacy.

BARNARD S. PROCTOR.

NEWCASTLE-ON-TYNE, *July* 1873.

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# PRACTICAL PHARMACY.

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## LECTURE I.

Introduction—Division of Subject—Temperature for Drying Vegetables—  
“Vital Zone”—Drying Closets—Rate of Evaporation at  $x^{\circ}$  Fahr.—Air  
Contains Aq.—Latent Heat of Steam—Evaporation over Oil of Vitriol—  
Use of Quick Lime.

PHARMACY may be defined to be the study of remedies. Like most other sciences or arts of a miscellaneous nature, it is surrounded by other sciences, more definite, and generally more abstract in their character, which take, piece by piece, large sections from the province of Pharmacy.

Materia Medica, Therapeutics, Chemistry, and Botany, combined into one art, formerly constituted Pharmacy, but these sciences gradually becoming separate, the term Pharmacy is now applied to such ill-defined and unclaimed ground as has not yet been appropriated by any of these more abstract sciences. The duty of the lecturer on Pharmacy is, therefore, to give instruction in all the manifold subjects connected with the preparation of remedies, which are not already taught, or not sufficiently taught by the lecturers upon these previously-named subjects. To this task I will now devote myself; and, in doing so, I shall have to take it for granted that my audience have acquired the ground-work of the sciences I have enumerated, and that it is unnecessary for me to explain in detail many of the

principles and laws upon which the operations under consideration depend ; but, at the same time, I shall not hesitate to treat of any branch which may be connected with my subject, though the same may also belong to the province of another lecturer. I shall be guided solely by the desire to make the subject clear, systematic, and comprehensive, and the information imparted practically useful. I shall keep in mind that I am addressing *students* ; and while I remember that their first object will be to qualify themselves for their examination, I will not forget that their ultimate object must be the acquirement of such pharmaceutical knowledge as will enable them to carry on their business with success to themselves and satisfaction to the public. I shall not offer any remarks upon the importance of my subject ; for I do not expect to address any who are not convinced of the utility of acquiring, by every means within reach, a thorough knowledge of the art which it is my duty to teach. It is not my desire to tempt those who would otherwise be indifferent to this study, but rather to help those who have put their hands to the plough, with the determination to be industrious cultivators of the province of Pharmacy.

I shall count upon my hearers knowing beforehand a great deal of what I may have to say, or to illustrate, but I shall also count upon the greater part of what I may say being new or useful to some one or more of those who may hear me ; for a subject is rarely so thoroughly known that an attentive listener will not learn something from the experience of another. Useful matter will bear repetition, whether I may be repeating the instruction gained from another lecturer, or from the practical experience of business, and any who may from time to time feel that I am repeating an oft-told tale, will remember that I am a fellow-student with themselves, and will find it not unprofitable to compare notes of my knowledge and experience with their own.

Whenever possible, the subject will be treated in a practical way, illustrating the matter in hand with diagrams, apparatus, and processes in operation ; and with the view of ascertaining

how far I am successful in my teaching, I shall from time to time hold examinations of my pupils, devoting part of an evening to questions upon the subjects treated of in several of the preceding lectures, and concluding with a recapitulation of the matter, with the view of enforcing any important points which do not appear to have been sufficiently apprehended.\*

Practical Pharmacy may be divided into several sections, and though perhaps not a strictly scientific division of the subject, I have thought it most convenient to treat it under the following heads:—

Abstract or General Pharmacy,  
Official Pharmacy, and  
Extempore Pharmacy.

It may at first sight appear inappropriate to speak of any branch of an applied science, or art, as “abstract;” nevertheless, it is convenient to gather together under this heading a group of facts, principles, processes, and appliances which are so far abstract that they may be common to a great number of occasions, and in great measure independent of the material which may at one time or another be subjected to our manipulation. Thus, grinding may be said to be an abstract process; which will at one time be applied to tartaric acid, and at another to jalap root. Infusing is an abstract process which will at one time be applied to calumbo, at another to senna,—and it will facilitate the treatment of the whole subject, if under the head of abstract or general processes, all those matters are discussed which have a wide applicability.

Under the head of Official Pharmacy will be treated the processes of the Pharmacopœia, arranged in such groups as naturally assimilate, rather than in alphabetical order, which would bring in contiguity many processes having no natural connection. The several operations described in the first section—Abstract Pharmacy—will then be exemplified, with the various modifications necessary to meet the requirements of special cases.

\* To the student reading these pages, it is recommended to follow the same system of self-examination.

Under the head of Extempore Pharmacy will be treated the operations of the dispensing counter, many of them bearing considerable analogy to the processes of the Laboratory, but so far distinct as to require a special experience for their quick, accurate, and satisfactory performance.

Reverting to abstract processes, they may be classified according to their objects, and the mode in which that object is to be accomplished.

*First*, The division of a substance into smaller particles of a like nature, and the separation of finely-divided from coarse particles, by such operations as cutting, bruising, grinding, sifting, and washing.

*Second*, The reduction of a solid body to the liquid state, by solution, and the separation of a substance into parts of different natures, as in the preparation of infusions, and the washing of precipitates, in which cases soluble are separated from insoluble matters—the one being used, and the other rejected.

*Third*, Consolidation by the dissipation of a solvent, leaving the solid of the same nature as it was in solution, including such operations as the evaporation of fluids to solid extracts, and the production of crystals from their solutions.

*Fourth*, The combination of solids of different natures into one mass, as in the production of pill masses. And this division will leave a considerable number of processes unclassified, including distillation, sublimation, calcination, &c. All these processes are essentially simple in their nature ;—they are elements out of which we have to construct the complex processes required for the preparation of many pharmaceutical products. In the second division, these elementary operations will be used together in various degrees of complexity, up to the elaborate processes involved in the production of some of the organic acids and alkaloids. We will commence with the reduction of massive materials into the state of powder.

Inorganic substances, such as native minerals and salts, may generally be submitted to bruising or grinding, without any preparation ; but articles of vegetable materia medica will

generally require to be dried, and we will first briefly consider the various appliances which have been devised for drying, together with general precautions requisite to avoid injury of the substance under treatment.

### DRYING.

In tropical climates, little else is necessary to insure satisfactory desiccation than exposure to the air and sunshine, but in our own humid climate that cannot be trusted. Warmth and moisture are the essential conditions of rapid change in organised bodies. There is a zone of temperature which may be called the vital zone, within which the functions of animal and vegetable life are possible. The freezing and the boiling points of water may be considered the limits of active life, though in some recent experiments by Crace Calvert, reported in the *Pharmaceutical Journal* of 2d September 1871, satisfactory evidence has been found that infusorial life is capable of withstanding a heat of 300° Fahr. in infusions contained in hermetically sealed tubes, capable of bearing such a pressure as is involved by subjecting watery fluids to this temperature. He experimented upon solutions of sugar and of gelatine, and infusions of hay and of putrid meat; and in all cases found that a temperature of 212° Fahr. was insufficient to destroy the infusorial life which had made its appearance by keeping those liquors in contact with the air; and that in all cases, except the solution of gelatine, they continued in diminished proportion even after 300° Fahr. had been reached but that in all cases a temperature of 400° Fahr. was sufficient to effect the total disappearance of vitality. According to these observations, the infusorial fungi are most readily destroyed, a temperature of 212° Fahr. being fatal to them; and the vibrios the least readily destroyed, their vitality continuing in solutions exposed to 300° Fahr., but not in those heated to 400° Fahr. He also found intense cold incapable of destroying these low forms of protoplasmic life, a liquor containing microzima and vibrios, on being frozen and retained for twenty hours at temperatures

ranging between 32° Fahr. and 15° Fahr., still displayed living animalculæ on being melted, and the movements, which were languid on the first thawing of the ice, resumed their wonted energy in the course of a couple of hours.

But though he has shown that the animalculæ did not lose their vitality, we have other evidence that the life was in a condition which we may call latent at the lower temperature, and is it not unlikely that the same may be the case when the temperature is above 212° Fahr.,—that functions are languid, if not entirely suppressed, and that vitality is potential rather than active. We will, therefore, continue to suppose that, as regards functional activity and the chemical changes which accompany it, the freezing and the boiling points of water may be regarded as the extreme limits of this zone, and the portion characterised by a marked activity is much more limited. Fermentation requires a temperature between 50° Fahr. and 90° Fahr. and is at its greatest activity between 70° Fahr. and 80° Fahr.; and though fermentation is not the only change to which moist vegetable matter is subject, it is a very important one, and the circumstances which promote it also promote other species of decomposition. Again, the boiling point of water comes in as a *maximum* temperature, to which vegetables may be submitted in drying, and there are many of them which will not bear this heat without injury. A little above a 100° Fahr. we begin to find changes which are liable to affect more or less the properties of the body subjected to it. At 140° Fahr. starch, in the presence of water swells, and becomes soluble. At the same temperature albumen coagulates; between this and the boiling-point, many vegetable substances of a complex nature also undergo changes. If, therefore, we set down 90° Fahr. as the lowest temperature to which vegetables can be continuously subjected in the presence of moisture, without undergoing fermentation, and 140° Fahr. as the point at which danger from over-heating commences, we have indications of the suitable range within which all ordinary processes of vegetable desiccation may be performed.

HEAT OF DRYING-CLOSET.

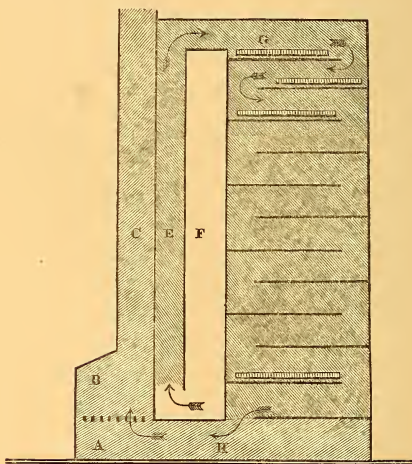
210	Zone.					
200						
190						
180						
170						
160						
150						
140		{	{ Starch dissolves.			
130			{ Albumen coagulates.			
120		{ Temperature for Drying Green Herbs.				
110						
100						
90	Vital	{	Fermentation	{ Fermentation active.		
80					{	Possible.
70						
60						
50						
40						
30						

It must not be supposed, however, that temperatures both above and below those bracketed are not on many occasions both safe and desirable. I have indicated 90° Fahr. and 140° Fahr. as the points within which almost anything may be safely trusted, and wish to impress upon you that the usual heat of sunshine in the open air is much too low for advantageous work. It simply brings the material into the zone of most active organic change, the conditions for the greatest perfection of vegetable life being also those of most rapid destruction when the vital action has been interrupted. Common herbs are frequently dried by being hung from the ceiling of a warm dry room, but where a large quantity has to be reduced to dryness in a short space of time, it becomes necessary to have recourse to special arrangements. In the drying-rooms connected with drug-mills it is usual to spread the herbs, &c., upon trays heated by steam-pipes, the waste steam from the engine which drives the mill being a cheap and convenient mode of applying a heat a little under the boiling point. The usual forms of drying closets, in addition to providing the heat requisite for evaporation, are so arranged as to cause a rapid current of air over the moist materials.



The accompanying sketch will show the arrangement. A is the ash-pit; B the fire-place; and C the chimney; E, a flue separated from the chimney and fire-place by iron plating, or brick work; F, a space open to the air; G, the drying trays; H, a flue from the bottom of the drying-closet to the fire. The arrows show the direction of the current of air. As the fire draws all its air from the closet, it insures a sufficient ventilation, and as all the air which enters the closet has to pass up the flue E, which is kept hot by its contiguity to the fire

Fig. 1.



and the chimney, its condition, being clean, warm, and dry, is most favourable for promoting evaporation. The cut represents the shelves arranged to cause a serpentine passage of the air over the trays.

Many modifications of this arrangement have been contrived to suit special conditions. I will only draw your attention to one, which I have devised myself, and which will probably be found suitable for the operations which most of you will have



to perform in the discharge of your duties as pharmaceutical or dispensing chemists.

It consists of a series of trays of thin iron-plate, so constructed as to pack away within one another when out of use, thus being convenient from its economy of space, especially for the use of those who have not daily occasion for its services. When put in operation, the lowest tray, which is also the smallest, is heated by a row of small gas jets placed at one end, at which end there are also perforations to admit air; the edges of the tray being about two inches high, the next tray may be placed upon it, leaving space enough for the moist material and the current of air which is to play over it. The second tray has similar perforations to those in the first, but placed at the other end, so that the air has to pass over the whole length of the first tray before it can ascend through the openings in the bottom of the second. A third, fourth, and fifth tray may be added, if required, taking care that the openings are in alternate ends, so as to ensure a serpentine channel for the heated air. I do not suggest this as an arrangement suitable for those who have desiccation continuously going forward, but for the occasional use of the pharmaceutical chemist, it will be found to have the following advantages :

It is packed away in small space when not required, and may be set in operation at a minute's notice. It is equally convenient for a single tray, or for two or three trays, full of material, according to the requirements of the operation in hand. The heat is easily regulated by increasing or diminishing the supply of gas.

Green herbs, such as conium, hyoscyamus and digitalis, should be dried at a temperature not exceeding 120° Fahr. It is, perhaps, to be regretted that the Pharmacopœia does not now give any instructions for the drying of articles of vegetable materia medica. But we have various authorities for stating that the temperature I have just indicated—120° Fahr.—should not be *exceeded* in the case of the more delicate materials, and as it is important that they

should not long remain warm and moist, it is desirable that the temperature should not be lower than is required to ensure their safety. On referring to the table\* of the rate of evaporation of water at different temperatures, it will be seen that it takes twice as long to evaporate a given quantity of water at 100° Fahr. as it does at 125° Fahr., and it takes four times as long to evaporate the same at 79° Fahr. Various other circumstances, however, have a great influence upon the rate of evaporation; most notably the percentage of moisture in the air, and the rapidity with which the air in contact with the materials is removed. Also the mechanical or physical condition of the material and its affinity for water;—thus aloes and opium take much longer to dry than senna or chamomile, because of their compactness, which impedes the escape of moisture; and alum parts less readily with its water of crystallisation than carbonate of soda, which may be equally compact, because of the former salt having a greater affinity for the water which it contains.

Under natural circumstances, air is never quite dry; it most nearly approaches this condition at low temperatures, but even at the freezing point 100 cubic inches of air will retain  $\frac{1}{100}$  of a grain of water. We may also say that every material has a certain affinity for water, and the relation between the affinity of a given material for moisture, and the percentage of moisture in the air will determine whether that given substance under these circumstances will lose or gain moisture.

*Exp.*—Two sheets of paper of the same nature in every respect, except that one is almost absolutely dry, and the other palpably moist, when exposed to the air, one will gain, and the other lose, moisture, provided the air is in anything like a normal

\* Relative quantity of water which evaporates during a given time at sundry temperatures.

At 212° Fahr. . . .	512	At 100° Fahr. . . .	32
180° „ . . . .	256	79° „ . . . .	16
150° „ . . . .	128	58° „ . . . .	8
125° „ . . . .	64	38° „ . . . .	4

*Redwood's Pharmacy.*

condition ; but the air might be so moist that the moist paper would not part with any moisture, or it might be so dry that the dry paper could not absorb any. Either of these latter conditions, however, would only continue so long as the temperature remained steady ; a fall in the temperature would enable the dry paper to absorb moisture ; a rise would cause the moist one to part with some. To illustrate this action with pharmaceutical materials, I weighed out two portions of senna from the same drawer ; each weighed exactly 50 grams (=771 grains) in the condition of approximate dryness which senna possesses under ordinary keeping. One of these was kept upon the drying trays for ten hours, the other exposed to the damp atmosphere of the cellar. Two portions of squill root of a like weight were subjected to the same treatment. During the ten hours drying the senna lost 58 grains and the squill 62 grains. During the ten hours exposure to the damp atmosphere, the senna gained 19 grains and the squill 10 grains. During three days exposure the senna had made a total increase in weight of 60 grains, and the squill of 30 grains. And to show that the gaining or losing of moisture by the exposure of a given substance to the air is determined by the relation which subsists between the degree of moisture of that substance and the moisture of the air, we now take these two samples of senna, the one palpably damp, and the other unusually dry, and expose them on sheets of paper to the air of the lecture room. An interval of half an hour having elapsed, the damp senna had lost ten grains, and the very dry senna had gained 10 grains, by exposure to the same atmosphere, under the same circumstances ; and no doubt a continued exposure there would in a short while result in the two samples becoming just equally moist—the exact degree of moisture ultimately attained being determined by the relation borne by the hygroscopic power of senna to the temperature and hygrostatic condition of the atmosphere. These experiments make no pretensions to precise accuracy, yet it is worthy of a passing note that the difference between the damp senna and

that which was dried till it became crisp, amounts to 118 grains of water, and that the squill root which we usually regard as having a remarkable affinity for water only differs to the extent of 92 grains. The fact that the squill, especially when powdered, becomes sticky if slightly damp, draws our attention to the fact that powdered squill absorbs moisture with avidity, a fact which we would probably overlook were it not forced upon our attention by the stickiness which is so troublesome, if the powdered squill be not protected from moisture with the most scrupulous care.

We have here a table showing the quantity of water which is capable of being held by a 100 cubic inches of air at several temperatures.

100 CUBIC INCHES OF AIR AT			
° Fahr.	Gr. Aq.	° Fahr.	Gr. Aq.
32 will retain	0·01	176 will retain	6·00
77       ,,	0·50	212       ,,	15·00
122       ,,	2·00	248       ,,	27·00

With regard to the last two figures, however, we must remark that 15 grains represents an atmosphere of steam only, at the ordinary pressure of the air, and 27 grains could only be contained in 100 cubic inches under a pressure of about two atmospheres, but that pressure would fail to bring any portion of it back to the liquid form so long as the temperature did not fall below 248° Fahr.

The quantity of water given in each case makes the air quite saturated, and it is only when far removed from this point that evaporation can be conducted at all advantageously. A glance at the table will show how enormously the rise of a few degrees increases the desiccating power of the air, and where economy of time is important, how great is the inducement to use as high a heat as possible; and since hot air soon becomes charged with moisture when in contact with wet materials, how important it is to keep up a constant current, so that there is always a thirsty atmosphere replacing the moist, before it becomes saturated. All this necessity for heat could not exist unless

the heat were in some sense "consumed," and in this instance it disappears in the form of latent heat. The quantity thus consumed in converting water into steam varies slightly, according to the temperature at which evaporation is taking place, but at the boiling point the quantity required to convert 1 lb of water into 1 lb of steam of the same temperature would be sufficient to raise it to 1178° Fahr., or to a bright red heat, had it been a fixed body incapable of converting sensible into latent heat; or, in other words, it would have sufficed to raise about 8 lb of water from 60° Fahr. to the boiling point.

It has commonly been stated that water requires the same quantity of heat to convert it into vapour, at whatever temperature the evaporation takes place, the quantity of latent heat of a vapour being greater at low, than at high temperatures; but more recent researches have shown that this is not strictly true, as will be seen by a reference to the table of latent heat of water vapour.

C = F.	Latent F.	Total F.	C = F.	Latent F.	Total F.
0 = 32	+ 1090	= 1122	150 = 302	+ 903	= 1205
50 = 122	+ 1027	= 1149	200 = 392	+ 840	= 1232
100 = 212	+ 966	= 1178	250 = 482	+ 774	= 1256

*From Regnault in Gmelin.*

Here you will observe that water evaporating at the freezing point, where it is only supplied with 32° Fahr. of sensible heat, absorbs 1090° Fahr. of latent heat, making a total of 1122° Fahr. But at the boiling point, where it acquires 212° Fahr. of sensible heat, it only renders 966° Fahr. latent, the total in this instance being 1178° Fahr. And that when boiling under high pressure, when it acquires 482° Fahr. of sensible heat, there are only 774° Fahr. rendered latent, the total increasing slightly with the rise in temperature at which the conversion into vapour takes place. This consumption of heat in evaporation causes considerable reduction of temperature in the air passing through a drying closet, except when the material is nearly dry, or of such a nature as does not permit rapid evaporation. The long serpentine course of the air is less advantageous when there is a considerable bulk of fresh herbs, the

evaporation from which is so rapid that air entering at as high a temperature as can be permitted, becomes cooler than is advantageous before it passes over the last tray.

Substances which are difficult to dry without injury are sometimes placed in vacuo over oil of vitriol. The absence of atmospheric pressure very much expedites the volatilisation of the moisture, and the oil of vitriol absorbs the watery vapour as fast as it is produced, thus maintaining the vacuum. This, however, is a refinement rarely used, except in analysis; and, in conclusion, I need only refer to one more method which is sometimes used for keeping vegetable substances in a state of thorough desiccation. It is more especially useful for such materials as are liable to become mouldy. The substance being introduced into a wide-mouthed bottle, such as is popularly known as a soda bottle, with a hollow stopper, the cavity of the stopper is filled up with quicklime, or other very hygroscopic substance. The material while in use is, of course, exposed a little each time the stopper is removed to enable a portion of the material to be taken out, but the moisture it thus absorbs is again abstracted by the absorption of the lime before it is likely that another portion of the drug may be required. This is very convenient for keeping powdered squill in good condition, which otherwise is so liable to become damp and sticky. It is also advantageous for the preservation of powdered ergot and other drugs, which are apt to be injured by animalculæ or mould. Thorough desiccation affords the greatest protection against this source of injury. The fact is, that like larger beings, the microscopic animals and plants find it very dry work living without water, so they die, and allow the ergot to enjoy its existence in peace.

#### QUESTIONS FOR SELF-EXAMINATION.\*

What changes do moist vegetables undergo by keeping?

\* The student is recommended, after reading a lecture, to test his knowledge of the matter by endeavouring to answer these questions, and then to compare his answers with the recapitulation which follows, or, if necessary, with the substance of the lecture itself.

At what temperatures do these changes proceed most rapidly ?

The most palpable change in a vegetable by drying being loss of water, what other changes may it undergo ?

What is generally the most desirable heat for drying vegetables ?

Does change in temperature make much difference in the rate of evaporation ?

What other circumstances besides change in temperature affect the rate of evaporation ?

What becomes of the heat used in drying a moist substance ?

Is the same quantity of heat lost in vaporising water at all temperatures ?

#### RECAPITULATION.

Herbs, &c., if kept moist, are liable to be affected by fermentation, mouldiness, and slow decay.

The two former of these changes are most rapid at about 70° to 90° Fahr., and may be considered to cease below 32° Fahr. and above 212° Fahr.

Vegetables, in being dried, lose water and volatile oils.

Some of their constituents may undergo oxidation, such as oils, resins, alkaloids, or acids. Starch may be rendered soluble. Albumen may be rendered insoluble.

The most generally desirable heat for drying vegetables is just above the fermenting zone, say 100° Fahr. to 120° Fahr. The rate of evaporation increases in a high ratio with increase of heat, but is also affected by the dryness of the air, and speed of its motion, by the pressure of the atmosphere, and by the compactness and hygroscopic properties of the moist material.

In converting water into vapour, about 1000° Fahr. become latent, but the quantity varies with the temperature at which evaporation takes place, the sum of the latent and sensible heat being 1122° Fahr. at 32° Fahr., and increasing a little as the temperature rises.



## LECTURE II.

### COMMINATION.

Slicing Fresh Roots—Cutting Hard Woods—Species—Rule for Degree of Commination—Bruising and Grinding—Steel Mills—Stone Mills—Sifting—Effects of Powdering—Experiment on Starch—Washing—Powdering Red Oxide of Mercury—Camphor.

ALMOST every solid substance used in medicine requires at some time to undergo mechanical division, either to fit it for direct medicinal use, or for some subsequent process. The nature of this mechanical division, and the means by which it is effected, will vary very much according to the physical nature of the substance to be operated upon, its crude condition before it is subjected to commination, and the degree to which the commination is to be carried. The substance after treatment will frequently not only have changed its mechanical condition, but also in some degree its chemical composition.

Our attention will now be given to the general objects of commination, the means of effecting it, and the changes of a chemical nature which are apt to be involved.

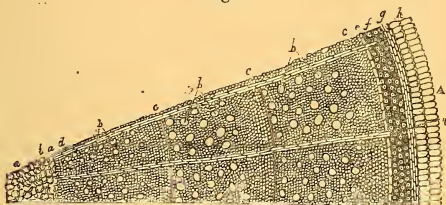
To commence with the most crude and simple operation, certain succulent vegetables, such as the bulb of squill and the root of calumbo, are sliced to promote their subsequent drying and preservation. To avoid the loss of juice a sharp instrument should be used, and to promote drying, the cutting should be transverse to the fibrous and vascular tissue of the plant. A blunt instrument by bruising the vessels will cause much loss of juice, and it must also be remembered,



that the transverse section, while it most promotes desiccation, also endangers the loss of juice more than a longitudinal division, which, while it may reduce the substance to equally small particles, does not to the same extent lay the vessels of the plant open.

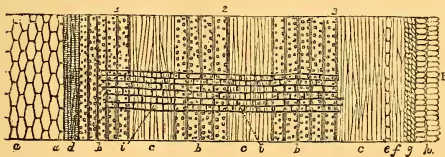
You will appreciate this difference, if you count the number of cells laid open in an equal surface of the two sections of a dicotyledonous stem, as figured below, the cuts being taken from "Bentley's Botany" p. 77, where you will find the structure fully described.

Fig. 2.



Transverse Section of a Dicotyledonous Stem three years old.

Fig. 3.



Longitudinal or Vertical Section of the same.

Some roots, such as gentian, taraxacum, and liquorice, are frequently kept sliced for convenience in making infusions or decoctions; in this case, as in the former, the roots are best cut transversely—as this section facilitates in the greatest degree the permeation of the root by the water.

In these instances the roots are usually cut dry, and most advantageously so, as this quite obviates any loss of juice, at

the same time the roots are not so bulky as to require division to promote their desiccation.

If, on the other hand, the object is to reduce a vegetable substance to a condition suitable for expressing its juices, the more completely the vessels are opened and torn asunder the better; but even in this case it will be readily seen, that a transverse section will most facilitate the expression of the juice. The instruments used in slicing vegetables are generally of the simplest possible nature, and are no doubt familiar to you all. Common pruning shears are very convenient for the smallest operations of this kind. There are sundry forms of root knives or choppers, which are suitable for working on rather larger quantities or more bulky materials, the object being to gain power by the use of a long lever, which constitutes the handle of the knife, while the short arm of the lever forms the hinge which fixes the knife to the block supporting the substance to be cut; the support is advantageously curved to prevent the root from slipping under the edge of the cutter.

With any of these appliances the task of reducing woody materials to slices is very laborious, and when required to be performed on the large scale, recourse is generally had to a lathe and turners' tools, as in the case of quassia, guaiacum, &c., but as this does not come within the province of the pharmacist, it is not necessary to enlarge upon the subject.

Dry substances which have been reduced by slicing, when compared with those which have been comminuted to an equal degree by other means, such as crushing in a mortar or mill, will be found to be more free from dust, consequently will more readily produce a clear infusion or decoction if required for that purpose. They are also more readily penetrated by the water.

It was formerly the custom to sell chopped herbs either mixed together or separate, under the name of species, from which herb teas, &c., were to be prepared; and though it is often convenient to have the materials for an infusion or decoction ready for its speedy preparation, it is not desirable

to have sundry ingredients mixed together, unless they can be suitably reduced to a rough powder, as materials in a *coarser* state of division—especially if part is fine and part rough—will not long continue to be uniformly mixed. If so fine a state of division is not desirable, the species should be put up in packets suitable for making a definite quantity of the resulting liquor—say a pint or a quart, each ingredient being weighed into each packet, to insure uniformity. The degree to which comminution is to be carried for any particular class of purposes cannot be fixed, but two or three principles may be stated which have a general bearing upon the question.

The more compact a material is the more minutely will it have to be divided to secure a satisfactory result.

The less soluble it is, or that portion of it is, upon which its activity depends, the higher state of division, *ceteras paribus*, will it require.

The less it is capable of expanding under the influence of the solvent the finer it should be comminuted. Thus vegetable substances generally swell more under the influence of water than under that of spirit, and consequently require to be more finely divided for making tinctures than for infusions, and as hot water causes a more rapid and complete expansion of the tissues than cold, a rougher condition may be used for a decoction or infusion with hot water than would be suitable for a cold water infusion.

Cinchona and cascarilla barks require finer division than gentian, calumbo, or quassia, both because their active constituents are less readily soluble, and because their structure is compact, and not freely penetrated by water or spirit used in making their preparations.

Materials which are to be administered simply suspended in water, or other vehicle, should be reduced *to the finest powder possible*. This last requirement brings us to the consideration of a different class of operations, viz., bruising, grinding, sifting, &c.

Operating on the small scale, as dispensing pharmacists will

generally do, bruising and grinding are both frequently done in the mortar by simple manual power, the process being so familiar as to require scarcely any notice. As I stated in my lecture on drying, thorough desiccation is usually an essential preliminary to the grinding of vegetable matter, and I only need now to refer to it as marking a difference in the nature of the coarse and fine comminution,—the former, effected by cutting, being usually performed with at least equal facility upon the fresh herbs, &c.; the latter, depending upon the brittleness and friability of the material, requires the removal of the water, which adds so greatly to the pliability and toughness of most organised structures.

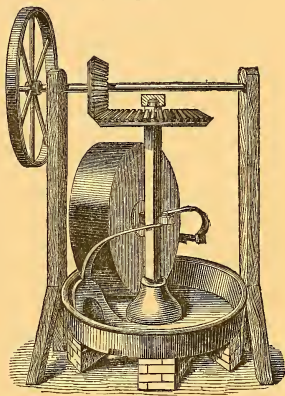
Steel mills are also very useful and convenient for grinding drugs where an impalpable powder is not essential, and they have the advantage of not requiring such absolute dryness as is necessary for grinding with stone, on which account preference should be given to them where the material contains active volatile matters, such as essential oils. The form so familiar as coffee or pepper mills will answer perfectly for a variety of purposes. It is essential to have them so constructed as to be readily and thoroughly cleansed between operations with substances of different natures. After grinding caraways or aniseed, the mill should not only be brushed out, but exposed to the air, to allow the evaporation of the essential oil with which it will have become impregnated, before it can be used for any other material. Steel mills may be considered applicable to vegetable substances only, while stamping mills, mortars, and stone mills may be used for every variety of materials. The stamping mill may be looked upon simply as a mortar beaten by machinery, and is frequently used as a preliminary to grinding under stones.

Stone mills are constructed upon two plans,—one being the form usually employed in grinding flour, consisting of two horizontal grindstones, the lower being fixed, and the upper revolving upon it. The material, in a rough state, but not in large lumps, being placed in a hole in the centre of the upper

stone, is drawn in by the motion of the stones, and torn to pieces, gradually becoming finer as it progresses from the centre to the circumference of the stone. As it leaves the stone it is in a powder varying in fineness according to the nature of the material, &c.; it may then be sifted, and the rough portions returned to the mill.

The other form of mill more commonly used in drug grinding, consists of one or two edge-stones running in a trough, the general arrangement of which will be best understood by reference to the diagram, taken from Redwood's "Practical Pharmacy," p. 82. The stones are necessarily thick, hard, and heavy, and usually run in a trough of cast iron. You will readily perceive that a stone such as that

Fig. 4.



shown in the diagram would run in a straight line if free from guiding power, and if forced round in a small circle, such as that it must describe in the trough, the side of the stone nearest the shaft will travel over only half the distance passed over by the other side of the stone, the side next the shaft traversing a small circle round the shaft, the off-side traversing a circle as much larger as double the thickness of the stone; but as both sides must necessarily revolve on their axis with equal velocity, some sort of compromise must take place between the rate of motion round its axis and the rate of motion over the surface of the trough. You will notice also that if a stone revolves either more rapidly or less rapidly than it progresses over the surface upon which it is rolling, there will be considerable friction.

If we suppose the stone to be a foot thick, there will be a

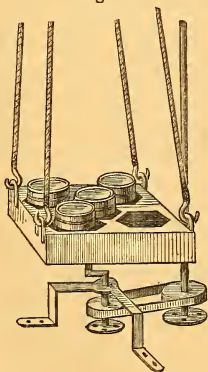
line on its edge, about half way between its two sides, that will revolve at about the same velocity as it progresses, and no one side of that line the stone will tear the material under it, in consequence of its revolving faster than it progresses, and on the other side of that line it will have the same action from revolving more slowly than it progresses.

The mere crushing force of the heavy stone rolling over the material would of itself reduce any brittle substance to powder, but tough and fibrous substances require a tearing action as well as direct pressure.

In grinding with the horizontal stones the weight of the upper stone is diffused over a large surface, but in the edge-stone the whole force and weight are concentrated upon a few inches.

It is said to be most economical, both of time and material, to let grinding and sifting alternate pretty frequently, and when the whole is finished, if the substance in hand be not homogeneous, it is necessary to mix the first sifting with the last, as the more friable portion which passed through first will frequently differ in nature or in the proportion of some

Fig. 5.



active constituent, from the more fibrous or tougher portion which has come through last. The sifting apparatus is usually the same as the familiar drum-sieve, and is kept in motion by the machinery which turns the millstones. A tray or shallow box, containing several drum-sieves, is suspended by ropes from its corners, attached to the ceiling. A crank attached to the bottom of the box causes it to revolve, not on its axis, but round a fixed point in the floor; and as a jerking motion promotes the sifting, advantage is taken

of the fact that a round sieve does not move smoothly if loosely fitted into a square hole. The cover of the box has angular



holes to receive the sieves, large enough to allow of their being shaken from side to side as the box revolves in the horizontal plane.

Other forms are sometimes used for articles the consumption of which is so large as to justify special sifting apparatus. A conical revolving sieve, such as used in flour mills, may then be adopted. It consists of a cone of wire gauze in a sloping position with the wide end higher. The sieve revolves in one direction, and a system of brushes fitting its interior revolve in the opposite direction. The material is continuously supplied to the wide end of the cone, and is brushed through as it runs down; only those portions which are too coarse to pass through run out at the narrow end.

A substance which is not homogeneous, either chemically or physically, if partially ground and sifted, the gruffs being returned to the mill and again ground and sifted, will yield powder of a different composition at the first from that obtained at the end of the operation, consequently, when the whole has passed through the sieve, the first and last portions should be thoroughly mixed together.\*

In some cases it is desirable to separate powders into particles of about uniform size, which is done by sifting through two sieves, the coarse sieve separating all the particles above a certain size, and a fine sieve separating all those below a certain size, there is thus obtained a sandy powder which is much less apt to become knotty than a powder containing particles of mixed sizes; but the practice is only admissible with homogeneous substances, as there might otherwise be produced a fine and a rough powder, differing not only in mechanical, but in chemical qualities.

It is convenient and unobjectionable in the case of sugar, which confectioners are in the habit of preparing and supplying to the pharmacist in three conditions, that known as medium powder being the sugar as it comes from the mill,

\* Compare instructions for the preparation of mixed powders in the "Pharmacopœia," p. 262.

having been sifted through a moderately fine sieve, and containing particles of all sizes, from the most minute up to the largest that are capable of passing through the sieve used. To obtain the fine and rough powders, a quantity of the medium powder is sifted through a sieve considerably finer than that it was first passed through. All that passes through this second sieve is known as fine powder, while that which will not go through is the quality known as rough powder; but besides the mere fact of its consisting of larger particles, the particles are much more uniform in size, and as a consequence, it is much less prone to agglomerating than either the fine or medium powders.

Mills and sieves, if their use be not confined to one material, will require thorough cleaning between the operations. Washing is objectionable, as the repeated wetting and drying is destructive of the machinery, and to avoid this it is a common practice to grind and sift saw-dust in small successive portions till it comes through clean,—a practice which is in itself unobjectionable, but which, it is said, led at one time to frequent adulteration of vegetable powders with the first portion of saw-dust which, having passed through the mill, came out pretty strongly impregnated with the material last ground.

The frequency with which powders are subject to chemical and microscopical tests will at the present time afford some protection against such practices.

The legitimate effect of powdering is, in the majority of cases, an increase in the strength from loss of water, and from separation of inert matter in the form of gruffs, such as the fibrous part of ginger root, the woody part of ipecacuanha, the husks of mustard and cardamom seeds, &c. Others, again, lose valuable volatile matters; powdered myrrh never makes so good an emulsion as the soft cheesy lumps of the crude article, and cinnamon, caraways, &c., lose essential oil. Saline matters are apt to lose water of crystallisation mainly in consequence of the drying which precedes the grinding. The disintegration



of starch, the coagulation of albuminous matter, the oxidation of some constituents, or the rendering insoluble of others, are changes which are liable to take place from the same causes, and more liable in drugs which are powdered than in those which are dried without being also powdered.

We have already considered the increased facility for extraction of soluble principles from vegetable substances, imparted by their division, but a few words more on that subject will be useful in connection with the changes produced by grinding. I shall have to treat of solution, diffusion, osmosis, &c., in a subsequent lecture, and cannot enter into these questions now, except just to point out that the whole vascular and cellular systems of plants contain deposits of one kind or other, some of which when in solution will readily pass through moist tissues, and others which, under the same conditions, will only pass with the utmost slowness. And there are some tissues which water will penetrate with the greatest facility, and others which are almost impenetrable, even when immersed for a long time. As I do not wish to anticipate the matter of a future discourse, I will only select one example, and demonstrate the facts by an experiment.

Starch consists of hard granules of matter soluble in water, covered with a skin of so hard and impenetrable a nature, that the starch in its entire state appears virtually unacted upon by water, and it may be rubbed in a mortar in the way you would rub any other material, with the view of crushing the agglomerations of granules, and yet each individual granule remains uninjured and insoluble, but if rubbed with strong pressure, the covering tissue becomes so far broken, that the soluble matter of the starch may be taken up by cold water, forming a slimy mucilaginous solution which, when filtered, may be tested with iodine, and will form the well-known blue solution, while the water filtered from starch which has been only gently rubbed does not give this reaction.\*

It rarely happens that any considerable number of the

\* Compare the tests for starch in the "Pharmacopœia."

granules are broken in grinding ordinary drugs (as may be ascertained by filtering cold infusions of powdered ginger, calumbo, &c., and testing them by the addition of a drop of tincture of iodine), but if the cells are broken which contain the starch,—and this is always the case in fine comminution,—it is much more freely dissolved in hot water than is the case if the material is roughly divided, and a comparatively small number of starch cells laid open. You all know how much boiling will be borne by barley without its being dissolved, though it is about 70 per cent. starch, but if the barley be first ground, the bulk of it will be dissolved by simply infusing in boiling water. Then we may take an illustration in which it is not desirable to dissolve the starch. Sarsaparilla root contains its active principles in the bark, and its farina in the pith, but as the latter renders the decoction no more active, but more prone to become thick and mouldy, the Pharmacopœia now directs the root be cut into convenient lengths, but not split open, as was formerly the custom, consequently we now have a decoction equally active, but less liable to decomposition than formerly.

For experiment, a sample of split sarsaparilla boiled a few minutes in water gave a copious blue precipitate with tincture of iodine, while a decoction of the unsplit root was not changed by the addition of this reagent.

The case of starch is probably an extreme one, but there are sundry mucilaginous principles which are not freely soluble nor freely diffusible when contained within the cells of vegetables, but which become readily diffused through an infusion if the comminution has been carried so far as to disintegrate the cells and expose their mucilaginous contents to the direct contact of the water.

There are some few instances in which a material may with advantage be ground wet; this was formerly done with shells, coral, calamine, chalk, &c.; but the use of the two former has died out, and the preparation of chalk does not fall into the hands of the pharmacist, so that the subject requires no

further notice than to allude to the process of washing, which in these cases takes the place of sifting. The powdered material being mixed with a large bulk of water, is allowed to settle for a short time ; the upper part, containing the matter in a fine state of division, is then decanted, and by long repose is allowed completely to deposit its contents, which are subsequently collected, drained, and dried, while the rough portion, which had subsided rapidly, is either rejected or submitted to a second trituration. In the case of chalk, of course the object is to get rid of sand, and the material being of little value, a considerable portion is thrown away, but when operating upon a valuable and homogeneous substance, such as the red oxide of mercury, the levigation may be repeated till every particle is reduced to a fine condition. Levigation on the small scale is performed either in a mortar or with a slab and muller. The minutiae of the operation can only be learned by practice, and will differ according to the material in hand ; the red oxide of mercury appears to me to grind best when only moist. In that condition it may be readily made so fine as to lose almost entirely the glistening appearance which it usually possesses, and until this is accomplished it is scarcely so fine as it should be for the preparation of eye ointments.

Singleton's ointment, which analysis has proved to contain simply red oxide of mercury and grease, appears to owe its superiority mainly to the careful powdering of the oxide. Chalk softens down best with a much more abundant supply of water. In washing, the quantity of water should be so large as not to acquire anything approaching a creamy consistence from the suspended matter, otherwise the separation of the rough particles will not take place freely. Camphor and spermaceti are best powdered by being sprinkled with spirit of wine before being rubbed in the mortar. There are some circumstances in which the use of spirit is not desirable in the powdering of camphor, and then it is well to remember that fixed or volatile oils and soap also have the effect of promoting its division ; and as they have not the volatility of

spirit, the powdered camphor obtained by their use is not so prone to agglomeration.

### QUESTIONS FOR SELF-EXAMINATION.

What are the principal varieties of comminution ?

What kind of materials are usually divided by cutting ?

In what direction are they best cut ?

What is the most notable difference between coarse comminution effected by cutting and by bruising ?

If species are kept for infusions, &c., what irregularity are they liable to introduce, and how should it be avoided ?

What circumstances determine the degree of comminution to which a substance should be subjected in preparation for maceration ?

What classes of substances are most necessarily ground in steel mills ?

What classes belong exclusively to stone mills ?

In what respects are powdered drugs likely to differ from those which have not been subjected to powdering ?

What substances grind better when not dry ?

### RECAPITULATION.

Comminution includes cutting, bruising, and grinding, the first being used for fresh vegetables and hard woods, which are most advantageously cut transversely, the product thus obtained being less dusty than that produced by bruising. Comminution should be carried further ; the more compact the material, the less it expands in the solvent in which it is to be macerated, and the more difficulty there is in dissolving the active matter. Comminution should be most perfect when an insoluble drug is to be administered in suspension.

Steel mills are advantageous for tough materials, vegetables

containing essential oils, &c. ; stone mills for such as can be dried till they become friable, and for salts and mineral substances. Powders are likely to differ from the massive drugs in having less water, essential oil, or other volatile matter, and in having less woody fibre or husk ; they are more ready to yield soluble matter to solvents, and are more liable to accidental or wilful impurity.

Red oxide of mercury is better ground damp. Camphor and spermaceti are most readily powdered by rubbing with a little spirit.

## LECTURE III.

### SOLUTION.

Classes of Solutions in British Pharmacopœia—Solution Defined—Solubilities Compared—Solvent Powers Compared—Effects of Temperature on Solubility—Presence of “A.” affects Solubility of “B.”—Freezing Mixtures—Rapidity not proportionate to degree of Solubility—Automatic Stirring—Supersaturation.

MECHANICAL division, if carried to its utmost, fails to convert a solid into a fluid. Yet we can scarcely look upon a solution as being other than a solid body divided into its ultimate molecules and mixed with the solvent. At the same time, many of the phenomena of solution are such as we do not readily bring within this simple mechanical theory. A consideration of these phenomena will afford us much interesting matter for thought, and the pursuit of our calling affords us many occasions in which all the information we can obtain relative to the theories or the facts bearing upon this subject will be acceptable.

In looking over the contents of the Pharmacopœia, we observe not only a variety of operations, but a variety of *classes* of operations depending upon solution, and our time will not be ill spent in glancing at each, with the view of estimating the subject-matter of our present study before we go into theoretical considerations.

First, there is a class of liquors, or “simple solutions,” in which a solid substance is dissolved in a liquid which does not alter its composition or properties, but will, on evaporation, leave it in the same condition as before it was dissolved.

Then there are cases of “chemical solution,” in which the

body dissolved enters into a more intimate combination with some portion of the solvent, and is not capable of being restored to its original condition by simple evaporation, as in the preparation of perchloride of iron or saccharated solution of lime.

Again, there are cases in which solution is the means of separating active from inert matter, as in the case of infusions, decoctions, tinctures, wines, vinegars, &c.

A fourth class, in which chemicals of different degrees of solubility are to be separated from each other by the subsequent processes of precipitation, crystallisation, or simple washing.

A fifth class, in which a liquid is made to dissolve a gas, as in the preparation of solution of ammonia, or solution of carbonate of magnesia.

And a sixth class, in which one liquid is made to dissolve another, as in the preparation of spirit of chloroform or liniment of mustard.

You will readily perceive how various must be the processes by which these operations are to be performed, how many circumstances will influence the production of solutions of these very different natures, and how often there is scope for the exercise of intellectual faculties, and the application of scientific laws in conducting these pharmaceutical processes.

Turning again to the point whence we started, we may first inquire into the nature of solution.

A solution is a fluid produced by the union of a fluid with a solid, a second fluid, or a gas—the union being so complete that the forces of cohesion and gravity do not cause separation, otherwise a mixture and not a solution has been obtained. The union must not be so intimate and powerful as to change the sensible qualities or combining powers of the solvent or dissolved body, otherwise a chemical combination, and not a simple solution has resulted.

In simple solution the taste, odour, colour, and combining powers remain the same, but in chemical combination they are generally quite changed.



In simple solution, if one, and only one of the materials used is volatile before the union, it continues to be so after the union, and may be separated by evaporation, leaving the other possessed of the same or nearly the same properties as before. Zinc will dissolve in diluted sulphuric acid, but the acid cannot be separated again by evaporation, because it has entered into chemical combination; but sulphate of zinc will dissolve in water, and the water may be separated again by evaporation, because the union has been only that of simple solution. And to illustrate the difference between solution and mixture, or suspension of a material in a liquid, the solution of sulphate of zinc may be set at rest for any length of time without subsidence taking place. Though the sulphate is heavier than the water, the bottom of the solution never becomes stronger by settling;\* had it been a mechanical mixture, such as might be made of hydrated oxide of zinc and water, a gradual subsidence would have taken place.

It is difficult to come to a satisfactory definition of solution, because of there being many exceptions to the sundry general rules which form our best guide to its comprehension; enough, however, has been said to give a tolerably correct idea of the matter.

I will first draw your attention to the solution of solids in liquids, and more particularly in water, for when we speak of a body being soluble or insoluble, the statement, if not otherwise indicated, has reference to water as the solvent. Its solvent powers are more extensive than those of any other fluid known. There are scarcely any chemical compounds except alloys, which we have not reason to believe are to some extent soluble in water, but the degree of solubility is ex-

\* In dense solutions which have been standing at rest in closed vessels for a considerable time, there will sometimes be observed a stratum of lighter liquid on the top, not resulting from any subsidence, but from the solvent having vaporised and recondensed upon the upper part of the vessel, and then run down again, and is found floating upon the heavier liquor. This is most frequently seen when a solution has been bottled while warm.



tremely various. We may take a few examples as illustrating this point.

Chloride of silver is soluble in 2,000,000 times its weight of water, but not in 1,000,000.

Sulphate of baryta is soluble in 800,000 times its weight of water, but not in 400,000.

Oxalate of lime requires about 500,000 parts of water.

Magnesia is soluble in 55,000 parts of water.

And sulphide of copper in 950,000 parts.

Looking to the other extreme, we find crystallised chloride of calcium is soluble in  $\frac{1}{4}$ th its weight of water, and fused chloride of zinc in  $\frac{1}{3}$ d of its weight of water.

So far I have spoken of the comparative solubility of various substances in one solvent, viz., water.

It is also interesting and instructive to compare the solubility of a substance in various solvents.

One part of camphor is soluble in—

1000 of water.

150 of water with magnesia.

1 alcohol sp. gr. 815.

$\frac{1}{3}$  of chloroform.

For experiment, I take a 4-ounce wide-mouthed bottle containing  $\text{ʒiij.}$  camphor shavings, into which I pour  $\text{fl. ʒj.}$  chloroform, which dissolves the great bulk of the camphor almost instantly.

*Exp.*—Gr. xx. each of camphor and calcined magnesia rubbed together, with  $\text{ʒiij.}$  of water, and filtered; the product is a concentrated camphor mixture without spirit, but containing magnesia.

Phosphorus is said to be insoluble in water, but is more or less soluble in various other solvents. *E.g.,*

One part of phosphorus dissolves in—

320 of alcohol.

20 of ether.

$\frac{1}{8}$  to  $\frac{1}{20}$  of bisulphide of carbon.

Showing that there is as great a diversity in the behaviour

of a substance with sundry solvents, as there is in the behaviour of one solvent with sundry substances.

It is a general if not a universal rule, that heavy solids when dissolved in water, add to both its density and bulk, the density of the solution being usually a safe guide to the percentage of the solid in solution. An illustration of this rule is found in Dalton's table of the density of solutions of caustic soda.

#### DENSITY OF SOLUTIONS OF SODA.\*

Aqueous Sol. Sp. Gr.	Contains per cent. NaO.	Boils at
2.00	77.8	Red heat.
1.85	63.6	315.56 C.
1.72	53.8	204.44 C.
1.63	46.6	148.89 C.
1.56	41.2	137.78 C.
1.50	36.8	129.44 C.

The addition of 0.1 sp. gr. indicating about 7 per cent. of NaO, the proportion being pretty constant throughout the table.

The table of density of acetic acid illustrates the exceptional cases.

#### ACETIC ACID.\*

Sp. Gr.	Per cent. of $C_4H_4O_4$ Glacial or Monohydrate of $C_4H_3O_3$ .	Sp. Gr.	Per cent. of $C_4H_4O_4$ Glacial or Monohydrate of $C_4H_3O_3$ .
1.001	1		{ 77
1.002	2	1.0732	{ 78
1.004	3	1.0735	{ 79
1.010	7		{ 80
1.015	10	1.0732	81
1.020	14		{ 82
1.025	18	1.0730	{ to
1.029	21		{ 90
1.040	30	1.0700	95
1.050	39	1.0680	97
1.060	50	1.0655	99
1.070	68	1.0635	100
1.0730	76		

\* Storer's "Dictionary of Solubilities," p. 454.

† Mohr, in Storer, p. 2.

There are two popular errors which I should wish to caution you against, which may be refuted by an examination of these and other tables of a similar nature, which you will find in your handbooks, more especially in "Storer's Dictionary of Solubilities."\*

I have heard it repeatedly stated that, if the density of a solution is expressed in relation to water as 1000, 1000 being subtracted from the density, the subtrahend expresses in grains the quantity of the solid in a fluid ounce. Now, if we calculate from the solution of soda, sp. gr. 1.50 (saturated at ordinary temp.), according to the rule given  $1.500 - 1000 = 500$  grains of soda in a fluid ounce. A fluid ounce of this density would weigh 656 grains, which, at 36.8 per cent., gives only 245 grains in a fluid ounce—the popular rule indicating in this instance double the true quantity. To make another calculation from the table of density of acetic acid—

$1050 - 1000 = 50$  grains in a fluid ounce; but a fluid ounce of this density will weigh 458 grains, at 39 per cent. indicated on the table will contain 178 grains of acetic acid. Thus the rule indicates more than twice the true quantity in one case, and less than a third of the true quantity in another. From an inspection of this table you will also observe that the addition of glacial acetic acid to water adds to its density, and even when the acid thus obtained has the same density as the glacial acid itself, which is the case when the proportion of real acid is about 65 per cent., a further addition increases its density. So that it is possible to mix together two samples of acetic acid, both sp. gr. 1063, and the result will be an acid of sp. gr. 1073. But a further addition of the stronger acid reduces the density again, and an addition of water would have had the same effect.

The other fallacy is, that an anhydrous salt when dissolved in water does not add to the bulk of the water, and that hydrated salts only add to its bulk as much as the bulk of the

\* A most valuable work, which the real student will find full of interest.

water they contain. To show the error of this statement, we will make a calculation from the table of densities of solutions of common salt.

CHLORIDE OF SODIUM SOLUTIONS.\*

Sp. Gr.	Per cent. of Sol.	Sp. Gr.	Per cent. of Sol.
100·725 . . . .	1	111·146 . . . .	15
101·450 . . . .	2	115·107 . . . .	20
102·899 . . . .	4	119·228 . . . .	25
105·851 . . . .	8	120·433 . . . .	26·395 (Sat.)
107·335 . . . .	10		

If we add 1 part of salt to 99 of water, it will yield a 1 per cent. solution, the density of which, according to the table, is 100·725; but if it had added nothing to the bulk of the water, its density would have been 101·010, as will be seen from the following calculation, the solution under these circumstances would of course measure 99, and weigh 100, its density being as  $99 : 100 :: 100 : 101·010$ . A similar calculation would give the density of a 20 per cent. solution as 125·000 instead of that obtained by experiment, 115·107. It is, therefore, evident that, whether we add 1 per cent. or 20 per cent. of salt to water, the bulk of the water is increased by the addition.

No satisfactory theory has been propounded by which to explain why one salt should be soluble, and another almost insoluble, in the same fluid. Silver, tin, zinc, and lead are very similar substances, and we cannot say why their chlorides should include the very extremes of solubility and insolubility in water.

But in some other cases we can trace what may be a rule in embryo, namely, the much more general solubility that exists between solids and liquids of a similar nature or constitution than that which exists between bodies of different natures. The solubility of oils, resins, fats, &c., in other oils, spirit, ether, &c., and their general insolubility in water, contrasts with the

\* Gerlach, in Storer's Dictionary.

generally sparing solubility of metallic salts in oils, ether, &c., and their generally free solubility in water. There are also some groups of substances having more or less analogy among themselves, which keep that analogy in their relationships to solvents. Thus the essential oils, having a general and physical analogy, much resemble one another in their degree of solubility in water and other solvents; and the alkaloids agree in being sparingly soluble in water, usually more soluble in spirit, and so on.

In the preceding cases we have spoken of solubility at ordinary temperatures, and we have next to consider the influence of change of temperature upon solution.

The general effect of a rise in the temperature is an increase in the solubility of solid substances, and a diminution in the solubility of gases. But there are sundry exceptions to the first section of this rule. Common salt is just about as soluble at the freezing point as at summer heat, though at the boiling point its solubility is slightly increased. Sulphate of soda increases in solubility rapidly from the freezing point up to 92° Fahr. (33° C.), about the temperature of the body, at which heat water will dissolve about five times its weight of the crystallised sulphate of soda,\* but the solubility decreases again as the temperature rises higher, till at 178° Fahr. (81° C.), when water will take up only about double its weight of the crystals; above which temperature the solubility again increases up to the boiling point—the weight soluble at this temperature being about  $2\frac{1}{2}$  times the weight of the water.

Hydrate of lime and sulphate of lime are also much less soluble in hot water than in cold, so much so that a saturated lime water will be reduced to half the strength on being heated to the boiling point without contact with air, the lime being deposited as hydrate.

*Exp.*—Lime water boiled in a flask deposits a crust on

\* 7 dry + 9 water = 16 xtl. salt.

the inside and on the surface of the liquid, the crust on the flask being crystalline, and much more visible when the water is poured out and the flask allowed to dry. This deposit will be hydrate of lime, and will dissolve in hydrochloric acid without effervescence if the lime water was free from carbonate; but as carbonate of lime is soluble to a small extent in lime water, it will generally be found that the deposit contains some carbonate.

Leaving the question of temperature, I will next direct your attention to the effect which the presence of one substance has upon the solubility of another. In cases where the solubility is increased, it is generally supposed that some chemical change takes place; but there are many instances in which that can scarcely be admitted. The well-known instance of corrosive sublimate being more soluble in the presence of alkaline chlorides, is explained by the fact that double chlorides of mercury and sodium, &c., are formed, which are more soluble than the simple mercuric salt. But the equally familiar example of the increased solubility of iodine in the presence of alkaline iodides cannot be accounted for in the same way; the iodine has every appearance of being dissolved, but not combined. It retains its dark colour and peculiar odour, and will volatilise and collect upon the upper part of the tube in its original condition. As a further experiment, we pour a solution of perchloride of mercury into a solution of iodide of potassium, and observe that the red iodide of mercury, which precipitates on the first contact of the liquors, dissolves into a nearly colourless liquid as it subsides through the solution of iodide of potassium. As soon as the latter becomes saturated with the former salt, we take a sample of the solution, and on evaporating obtain a pale yellow double iodide, showing that a chemical combination, as well as solution, had taken place. But, taking a further portion of the red iodide, we find it soluble in hot saturated solution of chloride of sodium, yielding a solution with no more red colour than the former, but from which it is again deposited in the form of brilliant red

crystals as the solution cools; thus the chloride of sodium appears to increase its solubility in hot water without forming a permanent compound.

There are many cases in which the increased solubility of one substance, imparted by the presence of another, is made practically useful in pharmacy, some of which will be noticed in future lectures. I will now only allude to one class of cases—that is, the production of freezing mixtures, where the production of cold, dependent upon the rapid solution of a large quantity of solid matter, is frequently attained by agitating with water a mixture of salts which are freely soluble, and which increase the solubility of one another. I shall have another word or two to say upon this matter presently in connection with the effects of solution. Returning to the subject in hand, the influence of one substance upon the solubility of another, I must briefly illustrate the reverse action to that which we have just noticed, namely, the *decreased* solubility of some salt in the presence of others.

Two or three of the most striking illustrations I can quote have occurred to me unexpectedly in my own operations.

Ordinary salt of tartar, though a very deliquescent salt, is almost insoluble in strong solution of ammonia, falling to the bottom like sand, gradually becoming moist, and after long contact forming a dense solution at the bottom, which does not mix with the lighter liquor of ammonia—

*Exp. 1.*—Put ʒiiss. liq. am. .880 and ʒss. salt of tartar into a 2-ounce phial, and agitate.

*Exp. 2.*—Put equal parts of saturated sol. carb. potass and liq. ammonia .880 into a phial, and agitate.

The dense solution containing a large quantity of potass and very little ammonia, the light solution containing the reverse, very little potass, and a large percentage of ammonia.

Ordinary pearlash, consisting of carbonate and sulphate of potass with small proportions of other salts, if mixed with its own weight of water and filtered, affords a solution of carbonate



of potass almost pure, the sulphate being insoluble in a saturated solution of the carbonate—

*Exp. 1.*— $\bar{3}j$ . pearlash,  $\bar{3}ss$ . water; rub together in a mortar, and filter off a portion of the solution; add excess of  $HCl$ , and then water and  $BaCl$ ; a very small precipitate takes place.

*Exp. 2.*—To the undissolved portion of pearlash of the last experiment add  $\bar{3}iv$ . or  $\bar{3}v$ . of water; filter off a portion; add excess of  $HCl$ , and then  $BaCl$  gives a large precipitate.

So decided is the action of the carbonate in this respect, that when saturated solutions of the two salts are mixed together, sulphate is precipitated.

*Exp. 1.*—Add  $\bar{3}j$ . saturated solution of carb. potass to  $\bar{3}j$ . sat. sol. sulphate potass, and stir together; a crystalline precipitate soon deposits, consisting of  $KSO_4$ .

*Exp. 2.*—Sulphate of copper thrown into strongest liq. ammon. gives a pale blue tint, very little of the copper being dissolved; add more water, and deep blue is produced.

*Exp. 3.*—To strong liq. ammon. add a little saturated solution of cupric ammonia sulphate, and a blue precipitate is formed, the supernatant liquor being pale blue.

These observations led me, through a long series of experiments, to various interesting results, which, however, are not important to our present subject. Some of them are recorded in the "Chemical News" of January 1864.

The rapidity of solution does not depend entirely upon the degree of solubility, some substances being very soluble, but slowly soluble; others dissolving more rapidly, but to a smaller extent.

Persulphate of iron may be taken as an illustration of a very soluble and deliquescent salt, which, when once reduced to dryness, is only slowly redissolved.

*Exp.*—To a test tube half full of water add a few grains of dry persulphate of iron, and agitate; a white milky liquid results, which very slowly changes to clear yellow brown (in the course of a few hours), but which rapidly undergoes the same change on being heated to boiling point.



Arsenious acid is another well-known example. If a few grains of it are shaken up in an ounce of water, and filtered off, the water remains almost uncontaminated with arsenic, though the ounce of water is capable of dissolving about 10 grains of the acid.

Whereas bitartrate of potass, which is only soluble to the extent of about  $2\frac{1}{2}$  grains in the ounce of water, dissolves so much more rapidly when treated in the same manner, that the water immediately becomes acid from the salt dissolved, and soon takes up nearly all it is capable of dissolving.

*Exp.*—Into a 2-ounce phial put  $\bar{3}$ j. aq. and 10 gr. of arsenious acid; shake, and filter off. The filtrate tested with cupric ammonia sulphate scarcely produces any precipitate. For comparison, have  $\bar{3}$ j. aq., containing  $\frac{1}{100}$  gr. arsenious acid, to which add some of the test.

Also—Into a 2-ounce phial put  $\bar{3}$ j. of water and 10 gr. of cream of tartar; shake, filter, and test with diacetate of lead, which produces an abundant precipitate.

When it is required to produce a saturated solution of any material, it is important to keep in mind the degree of rapidity with which it is dissolved, and that the speed of solution decreases very much as the point of saturation is approached.

If 100 parts of water will dissolve 100 parts of a given salt, the first 50 parts will be taken up much more rapidly than the second; and if the salt has just been thrown into the water and allowed to subside, the water at the bottom of the vessel soon becomes saturated, and consequently heavy; the supernatant water will float above for a long time, getting slowly impregnated with the salt. On the other hand, if the salt be supported near the top of the water in such a manner as to allow the water to circulate freely about it, that portion in contact with the salt becomes heavy and sinks, while its place is taken by a fresh portion of water, thus establishing currents, rapid at first, gradually becoming slower, and ultimately ceasing when the salt is entirely dissolved or the water quite saturated.

This kind of automatic stirring is constantly made use of in pharmaceutical operations and manufacturing processes; and wherever it can be made available, of course it saves the work of the operator.

*Exp.*—Two test-glasses full of water. Put into one half a drachm of permanganate of potass, which falls to the bottom; into the other suspend a similar quantity of the salt contained in a muslin bag; the stream of dark solution will be seen falling from the bag, the contents of this glass becoming highly coloured before the contents of the other glass.

When it is desired to estimate the solubility of a salt by the evaporation of a perfectly saturated solution, more careful operation is necessary. In most cases it is sufficient, if an excess of the salt be reduced to fine powder, triturated with the water for quarter of an hour, and the excess separated by filtration; but in the case of arsenious acid, or other substances which dissolve very slowly, several days may be required. Another mode is to employ heat, so as to dissolve more than the water can retain, and subsequently to separate by filtration that which has deposited on cooling; but even in this case it is necessary to use a long-continued trituration to insure the separation of the excess, for many solutions formed with the aid of heat will continue to deposit crystals for a long time after they are cold. Solutions in this condition are known as supersaturated solutions. The phenomena of supersaturation have been made the subject of many interesting papers, more especially by Mr C. Tomlinson. The well-known case of sulphate of soda gives us a familiar and useful illustration. I have already pointed out its great solubility at 92° Fahr. (33° C.); and it is well known that a solution saturated at this temperature will, under certain conditions, retain the whole of the salt in solution after it is cold, but the contact of a suitable nucleus suffices to change the solution into a mass of crystals.

*Exp.*— $\bar{5}$ vj. sulphate of soda in  $\bar{3}$ ij. of water, heated to 100 Fahr. till dissolved, filtered into a phial which has been

washed with liq. potassæ, and then with distilled water; the phial being corked while the solution is hot, and then set at rest to cool, the solution does not crystallise till supplied with a nucleus.

Mr Tomlinson's theory is, that the activity of the nucleus depends upon there being a greater adhesion between the nucleus and the salt than between the nucleus and the water, a point which is scarcely yet satisfactorily settled, but upon which it is unnecessary for me to enlarge at present.

## LECTURE IV.

### SOLUTION—*CONTINUED*.

Solution of Liquids—Difference between Solution and Mixture of Liquids—Solubility of Gases in Water—Woulf's Bottles—Effects of Pressure on Solubility of Gases—Effects of Temperature—Solution causes Changes in Temperature—Solution of Hydrated and Anhydrous Salts—Change in Temperature on Solution of Liquids and Gases.

THE phenomena of solubility among liquids are generally of a simple character, so far as they are at present known.

There are some liquids, such as alcohol, which will mix with water in all proportions; others which are soluble in water to a notable extent, but not in all proportions, such as ether; and a third class, such as oils, which when mixed with water almost entirely separate. The first class is frequently regarded as a case of simple mixtures, the term solution being applied only to those cases where there is a limit to the quantity of one liquid which another will take up, but this can scarcely be regarded as a philosophical restriction of the term. One of the distinctions between mixtures and solutions is, that gravity has the power of separating mixtures of fluids of different densities, but not solutions. We may add water to water, and regard it as a mixture of two portions of water, seeing that there is no appearance of the ordinary operations of the law of gravity being interfered with; but when water is added to alcohol, and gravity ceases to have the power of drawing the heavier liquid to the bottom of the vessel, we must acknowledge that there is something more than a mere mixture, and we appropriately say that the two liquids are intersoluble in

all proportions. We have many examples of these various degrees of solubility among liquids in the daily operations of pharmacy; the dilution of alcohol and the fluid acids being of the first class; the preparation of spirit of chloroform and of mustard liniment being of the second, chloroform being very soluble in rectified spirit, yet not soluble in all proportions; and the castor oil contained in mustard liniment being soluble in all proportions in absolute alcohol, soluble to a considerable extent in rectified spirit, and to a smaller extent in weaker spirit. Bodies in the fluid state are most favourably circumstanced for exerting their affinities for one another, consequently solutions are generally instantly produced when soluble liquids are mixed together.

Heat in many cases increases the solubility of one liquid in another, as in the case of castor oil and rectified spirit, or chloroform and spirit; for it must be observed that though chloroform is very soluble in rectified spirit, spirit is not very soluble in chloroform; 5 per cent. of spirit will not dissolve in cold chloroform, though it dissolves a little below its boiling point, and separates again to some extent on cooling. 50 measures of castor oil mixed with 50 measures of rectified spirit do not form a complete solution at 60° Fahr. (= 15·5 C.), but with a slight elevation of temperature they combine perfectly, and on cooling separate again into 12 parts of spirit containing very little oil, and 88 parts of a heavier solution in which the oil preponderates.

In observations of this kind, however, it must be noted that rectified spirit is a solution of alcohol and water, and a little variation in the percentage of water it contains has a most important influence upon the results obtained, both by influencing the combining power of the spirit, and from the liability of either the alcohol or water to be precipitated by the superior affinity of the other for the substance added.

The aromatic distilled waters are examples of the third class of fluid solutions, the water being capable of taking up only a small percentage of the oil.

The solubility of essential oils in water appears to be increased by distilling the two together, but it is not unlikely that there is some difference in the quality or composition of the oil dissolved by distillation from the material yielding it, and that dissolved by agitating with water the oil which had been obtained by a previous distillation. The odour of the waters obtained by agitating the oils of cinnamon, rose, or orange flower with distilled water, is not the same as that of the water obtained by the usual process of distillation.

A few drops of oil of peppermint agitated with an ounce of water, and strained through tow to remove the larger globules of undissolved oil, leaves a milky-looking water which becomes bright on the application of heat, showing the increased solubility of the oil in water at the higher temperature—a circumstance which it is well to bear in mind when we have occasion to extemporise an aromatic water.

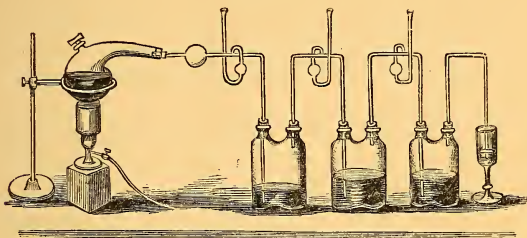
The solubility of gases in water is no less various than that of solids and liquids, and our pharmacopœial preparations afford us many examples—most notably the solutions of ammonia, nitric and hydrochloric acids.

The most condensable gases are usually the most soluble. The processes of dissolving gases are almost the reverse of those used in dissolving solids; the solvent is kept as cool as possible, and the gas is passed to the bottom of it, the supply of gas being regulated according to its solubility. The gas passing up in round bubbles exposes but a small surface to the action of the water, and from its rapid ascent it is exposed only for a very short time. Consequently, unless the solubility is extremely great, the bulk of the gas will escape unabsorbed, and to avoid this loss it is usual to employ a series of bottles arranged in such a manner that the gas which escapes absorption in the first bottle will pass in succession through the others. The gas in the first bottle is thus also subjected to a little increased pressure, which materially increases its solubility.

The following cut indicates the nature of the apparatus.

The tubes conducting the gas from the retort to the bottles, and from one bottle to the next, have attached to them bent

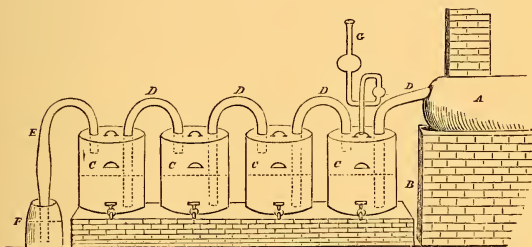
Fig. 6.



This Cut is taken from Cooley's "Cyclopædia."

safety tubes, into which a little liquid is poured so as to half fill the bulb. If a sudden evolution of gas takes place in the

Fig. 7.



Cut in Cooley's "Cyclopædia," page 153.

- A Iron retort.
- B Furnace.
- C C Stoneware condensers.
- D D Stoneware connecting pipes.
- E F Waste pipe and receiver.
- G Safety tube.

retort, the pressure forces this liquid up into the cup of the funnel and allows a little gas to escape, thus saving the



apparatus from any chance of bursting. If, on the other hand, rapid condensation should take place from cooling of the vessels, the liquor is drawn into the bulb, and air bubbles up through it, obviating the risk of the liquors being drawn back from one bottle into another, or from the bottles into the retort. In the manufacture of liquor of ammonia, large stoneware jars are used, and stoneware connecting pipes, as represented in fig. 7.

There is another expedient in use in large manufacturing operations by which to secure the complete absorption of gases. It is known as a coke column, and is more particularly useful in absorbing hydrochloric acid when mixed with air, and the products of combustion from alkali furnaces, &c. It consists of a large vertical flue filled with lumps of coke, down which water is constantly trickling. The coke being saturated with water, presents to the gases an immense surface of a highly absorptive character; the gases pass in at the bottom, and as they ascend are at every step brought in contact with water less and less impregnated with acid, till they escape from the top with scarcely a trace of acid unabsorbed. This process is called scrubbing, and the coke column is called a scrubber.

Passing a soluble gas through water at a given temperature and pressure, a definite strength of solution will be ultimately obtained; but if another gas be then passed through at the same pressure and temperature, part of the gas first absorbed will be given out again; and if a mixture of gases be passed through the water it does not become so completely saturated with either of them as when one only is present. This is a fact to be kept in mind in preparing solution of sulphurous acid. The pharmacopœial formula yields both sulphurous and carbonic acid gases; the latter gas interferes with the complete saturation of the water; a stronger solution is more readily obtained by evolving pure sulphurous acid from sulphuric acid and copper, or any of the other well-known methods.

Practically speaking, a gas subjected to double pressure is



reduced to half its original volume. This is not absolutely true of all gases and of all pressures, but for practical purposes the bulk of a gas may be said to be in inverse proportion to the pressure. Another rule regarding the solution of gases by water is, that at a given temperature the volume of gas which water is capable of dissolving remains the same, to whatever pressure it may be subjected.

For example, 100 cubic inches of water at ordinary atmospheric temperature and pressure will dissolve 100 cubic inches of carbonic acid, weighing 47·3 grs., but under a pressure of two atmospheres, 47 grs. of carbonic acid only measure 50 cubic inches; or in other words, it will require 94 grs. of carbonic acid to measure 100 cubic inches; and, according to the rule just given, it will require 94 grs. to saturate 100 cubic inches of water under a pressure of two atmospheres. But under greatly increased pressures, this rule ceases to be strictly applicable; thus it is said by Courbe, that under a pressure of seven atmospheres, only 500 cubic inches are dissolved; and it is well known, that when the pressure is increased till the carbonic acid liquefies, the fluid acid is not dissolved freely by the water. It is said, indeed, that under these circumstances the acid is only "slightly soluble in water;" but the difficulty of experimenting with fluids under these very high pressures probably prevents our information being reliably accurate upon this subject.

As a rise in temperature promotes the solution of solids, so a fall in temperature increases the solubility of gases. Under ordinary *atmospheric pressure*, water will dissolve its own volume of carbonic acid at 15° C. (= 59 Fahr.), but at 0° C. 100 cubic inches will dissolve 179 cubic inches of carbonic acid,\* provided the water remains liquid, but if it freezes the whole of the acid is expelled.

Bunsen's statement is perhaps not absolutely true regarding the total expulsion of carbonic acid, for the water obtained from melting ice renders solution of subacetate of lead turbid

\* Bunsen, in Dic. Sol.

from the presence of carbonic acid, though not to a much greater degree than recently boiled distilled water, as we may observe by an experiment.

*Exp.*—Ice water becomes milky on the addition of sub-acetate of lead;\* good recently distilled water less so. The same water boiled a minute gives a very slight haziness, and a sample boiled  $\frac{1}{4}$  hour gives none.

Under the same pressure, water will dissolve 727 times its volume of ammonia at  $15^{\circ}\text{C.}$ , or 1049 times its volume at  $0^{\circ}\text{C.}$ †

Carbonic acid dissolves more freely in alcohol than in water; ammonia dissolves less freely in alcohol than in water; gases, like solids and liquids, have their peculiar solvents, as well as particular conditions which facilitate their solution.

We may also note, in passing, that the experiment previously quoted, in which carbonate of potash was found to be sparingly soluble in solution of ammonia, also showed that ammonia was less soluble in water, which held in solution a large proportion of carbonate of potash, and now we add fused potash to strong liquor of ammonia (as the former is dissolved the ammonia is expelled), which is a convenient way of obtaining ammoniacal gas for experimental purposes. To illustrate another point or two, we pass the ammonia as it is evolved through two U tubes, the first being dry, and the second charged with small fragments of wet pumice stone. You will observe that the dry tube is not heated by the passage of the ammonia, but the first limb of the next tube becomes warm from the rapid liquefaction of the gas. The second limb of the second tube is not heated at all, and no ammonia escapes from the open end, showing how thoroughly efficient a “wet scrubber” is as a means of absorbing a gas, the whole of the ammonia being absorbed by the first three inches of wet pumice.

\* Lime water is not a sufficiently delicate test for carbonic acid to be used for this experiment, carbonate of lime being more soluble in the presence of the hydrate.

† Bunsen, in Dic. Sol.

A notable effect of solution is change of temperature. Solids in dissolving generally lower the temperature, the heat which disappears being consumed in their conversion from the solid to the liquid state. Gases, on the other hand, as we have just seen, when they become dissolved, part with the heat which was essential to their gaseous condition, and the resulting solution is warmer than the mean temperature of the gas and water before the solution took place. As a further experiment, which you may at any moment try for yourselves, a wet bulb thermometer placed in the neck of an ammonia bottle indicates a rise in temperature, from the ammonia condensing in the moisture which surrounds the bulb, and giving out its latent heat.

The solution of gases we have just noticed is promoted by reduction of temperature, and it will be readily seen that a larger quantity of heat being essential to a body in the gaseous than in the liquid condition, these two phenomena, the increased solubility at low temperatures, and evolution of heat on the rapid solution of a gas, become readily explicable; the molecular motion which constitutes gaseous elasticity and mobility, taking the form of sensible heat when the extreme elasticity and mobility, which are characteristic of a gas, become changed for the much smaller degree of these properties which belongs to liquids.

Solids, on the other hand, having less molecular motion than liquids, the conversion of the former into the latter necessarily requires the conversion of the motion of heat into the motion of fluidity.

The subject, however, is not quite so simple as it thus appears; the exceptional cases (where a salt is less soluble at a high temperature than at one somewhat lower, and where a salt in the act of solution evolves heat) require more careful examination before this theory can be accepted as universally applicable. In many of the exceptional cases the disturbing element is not difficult to discover. For example, anhydrous salts, when thrown into water, will commonly dissolve with the

evolution of heat; while the same salt, if first combined with water of crystallisation, would have dissolved with the production of cold. The solution produced by the action of water on an anhydrous salt must not be looked upon as a solution of the anhydrous salt, but of the crystalline hydrate, the heat evolved by the chemical union of the anhydrous salt with the water of crystallisation being, in some cases, of greater amount than the heat absorbed by the conversion of the crystallised salt into a solution.

This is a fact which must be remembered in the production of freezing mixtures; salts will produce a greater degree of cold if used in the form of crystalline hydrates than the same salt in an anhydrous condition. For example—

			Temperature falls from
Snow,* . . .	4 pts.	}	+ 32° to - 40° = 72° Fahr.
Dry chlor. cal.	5 „		
Snow, . . .	2 „	}	+ 32° to - 50° = 82° Fahr.
Cryst. chlor. cal.	3 „		

If we mix one equivalent (80 parts) of anhydrous sulphate of copper with five equivalents (45 parts) of water, the heat produced is sufficient to cause the ebullition of the water, the pale grey colour of the anhydrous salt being changed to the well-known blue of the crystalline hydrate.

*Exp. 1.*— $\zeta$ iv. anhydrous sulphate of copper thrown into a test-glass containing fl.  $\zeta$ ijj. of water.

But if we take one part of the crystallised salt and mix it with two and a half times its weight of water (the quantity requisite for its solution), a reduction of a few degrees in temperature will take place.

*Exp. 2.*— $\zeta$ j. fine powdered sulphate of copper; fl.  $\zeta$ iiss. water, mixed in a test-glass with thermometer.

These same phenomena may be observed with many salts, but I have selected sulphate of copper for the illustration, as it affords us additional evidence that the solution of a salt which crystallises in combination with water is to be looked

\* Gray's "Supplement to the Pharmacopœia," by Redwood.

upon as a solution of the *hydrate*, and not of the anhydrous salt, the blue colour of the solution corresponding with the blue hydrate, and being unlike the calcined sulphate, thus supplying a kind of evidence we cannot obtain with colourless salts. Had the experiment been performed with sulphate of soda, the phenomena would have been similar, with the exception that, being a colourless salt, we should have lost the benefit of the colour indication, and should have had to depend solely upon the difference of force with which the salt holds the two portions of water, for evidence that the solution is a solution of the crystalline hydrate, the water of solution evaporating with almost the facility of pure water (excepting concentrated solutions, especially of deliquescent bodies), leaving the salt in combination with water of crystallisation, which requires a higher temperature for its separation.

The temperature resulting from the dissolving of the anhydrous salt is probably determined by the relation between the affinity of the salt for the water of hydration and its affinity for the water of solution, and the proportion in weight which the salt bears to these two portions of water.

As a further experiment, we take three portions of sulphate of soda, the first with its ordinary water of crystallisation weighing 2 oz., the second dried at  $212^{\circ}$  till it has lost half its original weight (*i.e.*, now weighing 1 oz.), but still retaining one equivalent of water, and the third rendered anhydrous by a heat near redness, and adding each to as much water as is required for solution; note the change in temperature. To commence with, they all stand at  $57^{\circ}$  Fahr. before mixing, but on the addition of the salts, the first falls to  $47^{\circ}$  Fahr., the second rises to  $67^{\circ}$  Fahr., and the third to  $71^{\circ}$  Fahr.

In the solution of liquids there is a rise in temperature if condensation takes place, such, for example, as the solution of alcohol, or of oil of vitriol in water, the density of the solution being greater than the mean of the densities of the liquids before union.

If you examine a table of the percentage of monohydrated

sulphuric acid in acids of various densities,\* you will find evidence of the condensation which takes place on mixing the strong acid with water, thus—

100 vols. of acid will weigh 184, to this add half its weight of water, *i.e.*, 92 parts, and the volume of the mixture, if no contraction took place, would be 192, and its weight ( $184 + 92 =$ ) 276, and we should find as its bulk (192) is to its weight (276), so is 100 to its sp. gr. 142; but by reference to the table we find the actual density of an acid of this strength (66 per cent. of  $\text{H}_2\text{SO}_4$ ) is 1.55. Or to reverse the calculation, the sp. gr. of an acid of this percentage being 155, this number is to 100 as the weight of the mixture (276) is to its volume, in which way we find its volume to be 178, instead of 192. It has thus contracted rather more than 7 per cent. of its volume.

For experiment, we mix fl.  $\bar{3}$ j. of water with fl.  $\bar{3}$ ij of oil of vitriol in a small flask, and put a test-tube with  $\bar{3}$ j. of water into the mixture; the water shortly begins to boil.

Solutions of gases do not appear to obey a general rule as regards their density.

1. Solution of sulphurous acid is heavier than water, the liquid acid also being heavier.

2. Solution of ammonia is lighter than water, the anhydrous liquid ammonia being also lighter.

3. Solution of carbonic acid is heavier than water, though the liquid acid is lighter.

\* DENSITY OF SULPHURIC ACID OF DIFFERENT STRENGTHS.

Sp. Gr.	Per cent. of $\text{H}_2\text{SO}_4$ .	Sp. Gr.	Per cent. of $\text{H}_2\text{SO}_4$ .
1.8485	= 100	1.4860	= 60
1.8430	= 96.8	1.3884	= 50
1.8290	= 93	1.2999	= 40
1.8155	= 90	1.2184	= 30
1.7120	= 80	1.1410	= 20
1.5975	= 70	1.0682	= 10
1.5503	= 66	1.0074	= 1

More complete tables will be found in Attfield's "Chemistry," Gmelin's "Handbook," Storer's "Dictionary of Solubilities," &c.

### QUESTIONS FOR EXAMINATION ON SOLUTION.

What is solution?

What is the difference between dissolving zinc in diluted sulphuric acid, and dissolving oxide or sulphate of zinc in the same?

Name some of the salts most soluble and least soluble in water.

What effect has the presence of one salt upon the solubility of another?

Give examples.

What other circumstances have a great influence upon solubility?

When a substance is said to be very soluble, in what liquid do we understand that it will dissolve?

Does its solubility in water indicate its solubility in other liquids?

Is there any rule by which it can be predicted of a given compound whether it will be found soluble or not?

What is the general effect upon temperature caused by solution?

What is meant by a supersaturated solution?

### RECAPITULATION OF TWO LECTURES ON SOLUTION.

Solution is the union of a liquid (the solvent) with another liquid, a solid, or a gas, the combination being so intimate that gravity has not the power of separating the heavier from the lighter material, but not so intimate as to cause change in colour, odour, or taste, unless chemical combination as well as solution takes place. The chlorides of silver and zinc afford us examples of the extremes of solubility and insolubility, though chemically analogous.

The presence of one salt affects the solubility of another, in some cases increasing, in others decreasing it. Heat increases the solubility of most solids. Cold and pressure increase the solubility of gases. When solubility is spoken of, it is under-



stood to be in relation to water unless otherwise indicated ; solubility in water, however, is no guide to the solubility of the same body in other solvents. Bodies having analogous constitutions, and little chemical affinity, are usually most disposed to form simple solutions ; but no clear rule has yet been discovered by which to connect a body with its solvents. Simple solution of solids is usually accompanied by a fall in temperature ; solution of gases causes a rise in temperature. When a solvent retains more of the soluble matter than it could under the then existing circumstances dissolve, it is said to be supersaturated.



## LECTURE V.

### CRYSTALLISATION.

Crystalline and Amorphous Matter—Mother Liquor—Evaporation till Pellicle is formed—Separation of Heteromorphous Salts—Size of Crystals—Water of Crystallisation—Nuclei.

WHEN solutions are evaporated, or cooled, so much that the solvent is no longer capable of retaining the whole of the solid matter in solution, the latter is deposited in one of several distinct conditions. It may be that there is a glass-like residue, which is homogeneous, and devoid of any characteristic form, as is the case with gum, gelatine, citrate of iron and ammonia, &c., or the residue may have a pulverulent condition, as is the case with persulphate of iron (ferric sulphate) in which there are no distinct forms visible ; but, at the same time, the matter does not take the appearance of glass or jelly ; or, as in a third class, the residue takes peculiar and characteristic geometrical forms.

The two first are spoken of as amorphous conditions, the first being more characteristically so than the second. It is further distinguished as colloid. The peculiar properties of this class will claim our attention at a future time in connection with dialysis. For the present we will confine our attention to the matter which takes the crystalline form, or is deposited as a powder, in which traces of crystalline structure are frequently to be detected. The subject of crystallisation, taken as a whole, is much too extensive to be fully treated at present, and, in fact, belongs rather to chemistry and chemical physics than to pharmacy ; but there are several points to which it is desirable to draw your attention.

In most cases, when it is desired to obtain crystals, we avail ourselves of the difference in solubility occasioned by changes of temperature ; a salt which is more soluble at a high temperature being dissolved to its greatest extent in hot water, or other solvent, deposits the excess in the form of crystals on cooling.

SOLUBILITY OF SULPHATE OF COPPER.

100 of Aq. at	CuOSO <sub>3</sub> 5 Aq.	100 of Aq. at	CuOSO <sub>3</sub> 5 Aq.
0° C. dissolves	31·6	60° C. dissolves	77·3
10°        "	35·9	80°        "	118·0
20°        "	42·3	100°       "	203·3
40°        "	56·9		

If we have the salt originally in a solid state, but not crystallised, we may add to hot water as much of the salt as will dissolve, then on allowing it to cool we obtain a crop of crystals. The solution which has deposited them is called the mother liquor. This, on heating, may again be made to dissolve a fresh portion of the salt, which in its turn will be deposited as crystals on the solution again becoming cool. In this way the original salt may be taken up portion by portion till the whole is converted into crystals. But cases of this kind, where the sole object is to convert a mass of uncrystallised salt into crystals, are not of very frequent occurrence. It more commonly happens that a salt in process of manufacture is obtained in the form of solution, or is to be separated from some insoluble matter by being dissolved ; and, in this case the process preliminary to crystallisation is to reduce the quantity of water by evaporation, or boiling down, till the salt begins to separate in the form of a thin shell or pellicle on the surface. It may then be set aside to crystallise, and the mother liquor, which is subsequently drained off, will, on a further evaporation in the same manner, produce a second crop of crystals. In operating with salts which do not vary much in solubility with change of temperature, other methods must be adopted. Sea salt, for example, which is almost as soluble in cold water as in hot, is obtained in large crystals by the continued evaporation of sea water at ordinary temperatures, or is obtained in small crystals

fit for table use by boiling off the water with constant stirring, and raking up the deposited crystals from the bottom of the pan. In these three cases which I have selected to illustrate crystallisation in its simplest character, the salt has not been supposed to undergo any change beyond being brought into the crystalline state; but crystallisation is usually had recourse to as a means to some other end. Bodies which are not capable of crystallising are less confidently regarded as of definite composition, and bodies which are capable of crystallising lose one important evidence of purity if not obtained in this condition.

Certain groups of salts will crystallise in similar forms; thus the group of potassium salts, chloride, bromide, iodide, and fluoride, are of the cubic form. The group of alums, common alum, iron alum, chrome alum, whether they be compounds of ammonia or potass, crystallise in octahedra. These are called isomorphous groups, the meaning of the word being "of the same shape," but, as generally used, it is intended to express a similar chemical constitution, as well as a similar shape of crystal.

## ISOMORPHOUS GROUPS.

Magnesium.	Gold.	Chlorine.
Calcium.	Silver.	Iodine.
Manganese.	Sodium.	Bromine.
Iron.	Potassium.	Fluorine.
Nickel.	Ammonium.	Cyanogen.
Zinc.		
Cadmium.		
Copper.		
Chromium.		
Aluminium.		

If a solution containing salts, some of which are isomorphous and others not so, be made to crystallise, it will be found that the salts having a similar shape will crystallise together; those having a different shape crystallise separately.

To illustrate this, we will take the following experiment—

Common alum,	.	.	.	.	2 oz.
Sulphate of copper,	.	.	.	.	2 oz.
Iron alum,	.	.	.	.	$\frac{1}{2}$ oz.
Water,	.	.	.	.	8 oz.

Dissolve with heat, set aside in a beaker containing a slip of wood; as it cools crystals deposit upon the wood, which may be washed with a little cold water and tested. They contain iron and alumina, but not copper.

The mother liquor evaporated to a small bulk, and cooled, deposits crystals of two kinds,—sulphate of copper, containing traces of iron and alumina, and crystals of alum, containing abundance of iron, but only traces of copper.

Crystallisation thus becomes an important means of separating salts of one group from those belonging to other groups, though it is not nearly so advantageous in separating from one another salts belonging to the same group. Supposing alum to be crystallised from a solution containing sulphate of copper, the crystals may contain a small percentage of the copper, the great bulk of it remaining in the mother liquor; the crystals being again dissolved and crystallised, the second crop will contain still less of the copper, the second mother liquor having retained the greatest part of what escaped the first,—a second crystallisation frequently being sufficient to convert a crude commercial salt into one of sufficient purity for medicinal use, and a further repetition of the process will remove every trace of impurity.

The size and regularity in shape of crystals is increased by a very slow deposition; consequently, when these points are aimed at, the cooling of the solution must be very gradual, or spontaneous evaporation must be had recourse to, and agitation avoided. If, on the other hand, it is desired to have the salt crystallised in a granular or sandy condition, rapid cooling with agitation, or rapid evaporation with ebullition, is most successful.

It is frequently observed that salts crystallise with different proportions of water, according to the circumstances attending their crystallisation. Thus ordinary carbonate of soda in the usual course of manufacture crystallises with ten equivalents of water; but if the liquor from which crystals are to be obtained be completely saturated at the boiling point, the salt

is found to contain only eight equivalents of water. Then again, if solution of proto-sulphide of sodium be exposed to the air, carbonic acid is absorbed, and a carbonate of soda crystallises out, containing six equivalents of water; a carbonate with five equivalents of water is obtained in crystals deposited from the ordinary carbonate fused in its water of crystallisation, and a little variation in the temperature and method of operating will yield a fifth hydrate containing only one equivalent of water of crystallisation.\*

Sulphate of soda usually has ten equivalents of water, but it will also crystallise with eight equivalents, and even without any water at all. Proto-sulphate of iron usually has seven equivalents of water, but crystals may be obtained with four, three, or two equivalents.

Subjoined is a table showing the water of crystallisation in several common pharmaceutical salts.

WATER OF CRYSTALLISATION.

$\text{Al}_2\text{O}_3$	$3\text{SO}_3$	$\text{KO}, \text{SO}_3$	$24 \text{ Aq. } \%$ 45.5
<u>52</u>	<u>120</u>	<u>47 40</u>	<u>216 = 475</u>
$\text{CuO}$	$\text{SO}_3$	$5 \text{ Aq } \%$ 36	
<u>40</u>	<u>40</u>	<u>45 = 125</u>	
$\text{FeO}$	$\text{SO}_3$	$7 \text{ Aq } \%$ 45.3	
<u>36</u>	<u>40</u>	<u>63 = 139</u>	
$\text{MgO}$	$\text{SO}_3$	$7 \text{ Aq } \%$ 51.2	
<u>20</u>	<u>40</u>	<u>63 = 123</u>	
$\text{NaO}$	$\text{SO}_3$	$10 \text{ Aq } \%$ 59.9	
<u>31</u>	<u>40</u>	<u>90 = 161</u>	
$\text{NaO}$	$\text{CO}_2$	$10 \text{ Aq } \%$ 62.9	
<u>31</u>	<u>22</u>	<u>90 = 143</u>	

When a solution is sufficiently concentrated to crystallise, it will frequently happen that it is some time before crystal-

\* Gmelin, iii. 81.

lisation will commence, it may then begin without any evident cause, and go on rapidly to completion, or it may go on very slowly. It will frequently happen in large manufacturing operations that the growth of the crystals will continue for several days, or even several weeks, according to the nature of the salt, the size of the vat, the temperature of the season, &c.

The commencement of crystallisation is facilitated by the presence of rough foreign bodies, or particles of crystallised salt at the time when crystallisation ought to commence, and the completion of the process is expedited by increasing the surface upon which crystals can be deposited. This is sometimes accomplished by suspending twigs or strings in the solution. Crystallisation may sometimes be set going by friction, as in rubbing a stirring-rod upon the side of a beaker of liquor, the crystals depositing in lines which the rod has traced upon the glass.

A crushed salt, or a quantity of salt consisting of small and large crystals, mixed with a small quantity of solvent, will, after a lapse of time, become considerably changed, the small particles gradually disappearing, while the large masses increase in size. This takes place more rapidly with frequent variations in temperature. As the heat increases, the small particles dissolve more in proportion than the large, but as it cools again, the large grow at least as quickly as the small. It is probable that the result is also influenced by other causes, which, however, have not yet been sufficiently investigated. The phenomenon will be sometimes observed in dispensing a mixture containing more of some powdered salt than it is capable of dissolving, the salt, though finely rubbed when the mixture is made, becoming rough and knotted if the mixture is long kept.

## LECTURE VI.

### PRECIPITATION, ETC.

Crystalline Precipitates—Pulverulent Precipitates—Circumstances modifying Condition—Washing Crystals—Centrifugal Drier—Drying by Evaporation—Water of Decrepitation—Washing of Precipitates, by Decantation, by Filtration—Drying, by Pressure and Evaporation.

CRYSTALLISATION is sometimes produced by the mixture of two solutions of freely soluble salts, where decomposition results with the production of a salt which is not soluble enough to remain in solution, but is yet far removed from absolute insolubility. We have an instance of this kind in the formation of ammonia alum, by the addition of solution of sulphate of ammonia to a solution of sulphate of alumina, the alum being deposited in octahedra of a palpable size, because, though much less soluble than the two sulphates of which it consists, it is yet not so insoluble as to be separated instantly as an impalpable powder.

Another illustration is found in the granular sulphate of iron of the Pharmacopœia, which is deposited from a solution of ordinary sulphate of iron on mixture with spirit, the salt being considerably less soluble in a weak spirit than in water. It is, as in the last case, deposited quickly, but not so instantaneously as to fall in the form of impalpable powder.

We may thus, by gradual steps, pass from cases of well-marked crystallisation to indistinctly crystalline precipitates, such as carbonate of magnesia, to chloride of silver and other curdy or flocculent precipitates, which show no signs of crystallisation, but retain some property which causes their agglomeration, and to sulphate of baryta or chloride of mercury,



in which we lose all trace of their being other than amorphous and impalpable powders. But though the minuteness of the particles of which a precipitate is composed will depend in great measure upon its composition, it is quite possible to influence the mechanical condition in which a given chemical may be deposited. The more gradually a precipitate is thrown down, the more crystalline it will generally be found ; but even after deposition, long continued contact with the mother liquor may cause its change to a more granular or crystalline condition, and this generally takes place in a still more marked degree with the application of heat, especially with active ebullition.

When the physical condition of precipitates is in other respects unimportant, the granular state is preferred as facilitating their separation from the mother liquor, and their purification by washing.

The presence of foreign salts, which do not evidently increase the solubility, will sometimes increase the magnitude of the particles. I have obtained carbonate of magnesia, for example, as a heavy precipitate, almost sandy in texture, by adding a little carbonate of soda to a saturated solution of common table salt, which, as usual, contained chloride of magnesium as an impurity.

The washing of crystals, which is sometimes necessary to remove the mother liquor, and any impurities it may contain, must be performed with as little water as possible, to avoid loss of the salt; and before commencing, any clumps of crystals should be broken up, and the salt drained in a funnel till no more mother liquor drops out; then small successive portions of cold water poured upon the top, and allowed to drain between each addition, till it is found that the washings come away nearly free from the impurity which they are designed to remove. The appearance of the crystals is injured a little by this treatment, from the water dissolving off the sharp edges; and if it is desired to avoid this, the crystals may be washed with a cold saturated solution of the salt under treatment. It is not often practicable to use pressure to

remove the mother liquor, except in such cases as the crystallisation of crude hydrochlorate of morphia or aloin, in process of manufacture, the mother liquors in these instances, being syrupy, are removed from the crystals by pressure in a cloth, and the appearance of the semi-crystalline cake so obtained, being a matter of no moment, as it is to be redissolved, and again crystallised; but in many manufacturing processes, where the appearance of the product is a matter worthy of consideration, the removal of the mother liquor by means of centrifugal force may be adopted with the greatest advantage. In refining sugar, for example, the crystals of moist sugar are deposited in treacle, a fluid so viscid that subsidence and draining cannot be used for their separation; and the crystals being exceedingly soluble, washing is out of the question. The semi-fluid mass of treacle and sugar is thrown into an iron drum with perforated sides, which is made to revolve on its axis with great velocity, by which means the treacle flies off on all sides, leaving the sugar only slightly moist. The same mode has recently been adopted for drying Epsom salts, and after this treatment they are said to retain only from  $\frac{1}{2}$  per cent. to 1 per cent. of mechanically attached water, a quantity so small as to obviate any necessity for drying by exposure to the air or heat; the salt thus treated is more glistening in appearance, and quite free from efflorescence. In the case of Epsom salts, these advantages would not be sufficient to determine the use of the centrifugal drier, were it not that economy of time and money are also in its favour. There is no doubt that it might be much more extensively used with advantage in pharmacy, as it is free from the chemical action which is frequently objectionable in the use of heat or exposure to the air, and in its application to crystalline substances, is both more effectual, and less injurious to their appearance than mechanical pressure.

The appliances described as used for drying herbs, &c., in a former lecture, may be used for drying crystallised salts; but the operation is one of considerable nicety,

the precise degree of heat, &c., requiring adjustment to suit the properties of each salt; some, which contain much water of crystallisation, are apt to undergo aqueous fusion, with a slight elevation of temperature. This is the case with crystallised carbonate of soda; the bicarbonate, on the other hand, is apt to lose part of its carbonic acid. If it is desired to retain the crystalline condition of efflorescent salts, they must be dried at a low temperature, and removed from the drying trays as soon as a sufficient degree of dryness has been reached; but if it is desired to remove their water of crystallisation, the temperature, low at first, may be gradually raised, taking care that it never rises so high as to cause fusion. Deliquescent salts should also be dried with a gradually raised heat, to avoid their solution at a high temperature in the water, which was only mechanically adhering at a low one. A large number of salts are called permanent, being neither efflorescent nor deliquescent, and may be dried with less difficulty. A fourth class, such as the chlorides, iodides, and bromides of potassium, &c., have no water of crystallisation, but water mechanically confined between the plates of which the crystal consists; the water thus confined does not evaporate; but if a strong heat be applied, the crystals burst open from the steam generated. It is consequently called water of decrepitation; and, to obtain the salt free from it, the crystals should be roughly powdered before drying. They will then dry without difficulty, and are not liable to injury by any ordinary heat.

Turning our attention now to the treatment of precipitates, we find the difference in their properties from those of crystallised salts is one rather of degree than of kind. They are in particles of a smaller size; they are all soluble, but to a much smaller extent; they less commonly contain water of crystallisation, but frequently water of hydration, and generally much more water mechanically attached; and the salts contained in the mother liquor from which they have been thrown down will frequently adhere with considerable pertinacity. These facts determine the mode of treatment applicable to them.

Before proceeding to the washing and drying of precipitates, we must briefly note that precipitates will sometimes vary, either in mechanical condition or chemical composition, from very trifling variations in the mode of operating. The results will differ according as the solutions are hot or cold, concentrated or dilute, neutral or otherwise, and even according to which solution is poured into the other.

In illustration of these points, I may quote the precipitation of iodide of lead from its acetate by iodide of potassium, as described in Gmelin's "Handbook of Chemistry."

When strong and cold solutions of iodide of potassium and acetate of lead are mixed, pulverulent iodide of lead is precipitated. If the solutions are hot, or considerably diluted, the iodide is deposited in thin yellow laminar crystals. If the lead solution be poured into the iodide, the lead salt never being in excess, a pure iodide of lead is precipitated. If the iodide be poured into the lead solution, the lead is necessarily in excess at first, and an immediate precipitate of oxyiodide of lead is deposited. If the iodide of potassium solution be slightly acidulated with acetic acid, and dilute solution of acetate of lead added, fine crystals are deposited; but if the acetic acid be present in too large quantities, no crystals are deposited. If acetate of lead be dissolved in acetic acid and added to solution of iodide of potassium, dark-coloured iodide of lead is precipitated, containing excess of iodine.\*

I have not been able to verify this last result, and am inclined to think it an error; probably nitrate of lead, with excess of nitric acid, would be the correct reading of the note.

There are two methods of washing in general use,—first, by decantation, the precipitate being allowed to subside in the liquor from which it has been thrown down; water is poured upon it, agitation, subsidence, and decantation being repeated till the washings contain only an unimportant quantity of the impurity. In this process the most important points to be

\* Gmelin's Handbook.

noted are, that the precipitate must be thoroughly agitated with each successive portion of water; that the more completely subsidence and decantation are effected the sooner the precipitate will be clean; that a precipitate frequently contracts considerably in bulk as it becomes clean; that hot water allows subsidence to take place more rapidly; that it acts as a more powerful solvent of the matter to be washed away; that it promotes the aggregation of the precipitate; and, whenever it is admissible, effects the washing more quickly and satisfactorily than cold. The water may be decanted either by simply tilting the vessel, or by the use of a siphon. In the former case, for small operations it is convenient to grease the rim, and use a guiding rod to convey away the liquor, and prevent its running down the side of the vessel. The guiding rod is most suitably of glass, and is to be held in a vertical position against the greased rim, just in advance of the liquor which is about to be decanted. As the liquor is poured out, it will then run down the rod instead of dribbling down the side of the vessel, and is readily guided into any receptacle without loss. Washing by decantation is most suitable for precipitates which settle rapidly, and are of small bulk. Light and bulky precipitates are apt to flow over with the washing liquors, and in any case it is a prudent precaution to pour the washings through a filter, upon which the precipitate itself may subsequently be collected and drained. The second method of washing is by filtration, which is suited for precipitates which are light, bulky, and do not settle well. In operations on the smallest scale, the most suitable arrangement is the ordinary paper filter; and though this must be familiar to you all, there are one or two points to which it is desirable to draw your attention. The paper should be so folded as to fit the funnel nicely. The paper being folded in half, first in one direction, and then in the other, and opened out into the shape of a funnel, will, if folded accurately, always have exactly the same angle; but as funnels vary a little, it will sometimes be found that the paper fits tightly into the throat of the funnel, and is loose about the

top. This retards filtration, and risks the rupture of the paper, to avoid which, when the funnel is found to require it, the second fold of the filter paper should be made not exactly at right angles with the first; the paper may then be opened in either a more obtuse or more acute angle as required, and so adjusted as to suit any funnel that may be in use. I prefer to have the paper *slightly* more obtuse than the funnel. The paper having been adjusted, should be wetted before the precipitate is put upon it; this, causing the fibre of the paper to swell, prevents the precipitate from passing through, and also prevents the paper from becoming choked, as it would be if the pores were first filled with the precipitate, and then contracted by the swelling of the fibre. The wetting of the paper thus promotes both clear and rapid filtration. In this mode of washing, as in the last, it is desirable to use hot water whenever admissible; the rate at which it passes through a filter is in a very notable degree more rapid; and many precipitates which pass through the paper, if filtered from a cold solution, are effectually kept back if the solution and washing waters are hot.

For experiment, add a little chloride of barium and sulphuric acid to cold water, and filter the liquor; some of the sulphate of baryta will almost certainly pass through the paper. This, however, will be less the case if hot water be used, and may be entirely prevented by using actual ebullition.

For larger operations paper cannot be suitably used, and calico, flannel, or felt may be adopted, the former being most generally useful. It should be washed before it is used, both for the reasons given with regard to paper, and to remove the dressing which it usually contains when new. This is especially important if the filtrate is required for use, as well as the precipitate. The stand for a calico filter is made in the form of a four-legged stool inverted, the cloth being fastened by its four corners with strings to the parts representing the feet of the stool. Calico being more open in texture than paper, most precipitates will pass through it more or less at the com-



mencement of the filtration; but if the turbid filtrate be returned to the filter to avoid the loss of precipitate until the filtrate comes through bright, the washing may be continued without further difficulty. A precipitate having been left at rest upon the filter till no more filtrate drains out, is still imperfectly drained; but the dropping will begin again if it be gently shaken by striking the side of the funnel or filter bag; and when it ceases to give up water in this way, a further portion may be extracted by pressure. The paper filter may be taken out of the funnel, and carefully opened out flat upon a cloth, or a second sheet of filtering paper. The filter having been entirely unfolded, the precipitate will be found upon one half of the paper, and the other half may be folded over to cover it, while a dry paper or cloth is laid upon the whole, and gentle pressure employed to extract the water. It is desirable that the pressure be very gentle at first, or the paper will almost certainly burst; but as the water is expelled, the precipitate becomes so firm that the pressure may be increased to any extent, changing the outside paper or cloth till the precipitate is almost dry. To complete the desiccation, recourse may be had to the ordinary drying closet or hot-plate. The porous red-ware saucers used for putting under gardener's flower pots are convenient for drying precipitates in, as their porous nature permits the absorption of moisture and its evaporation from the whole surface of the saucer; but as the porous ware is difficult to clean, a saucer having been used for one material should not be used for another.

#### QUESTIONS FOR EXAMINATION ON CRYSTALLISATION AND PRECIPITATION.

What are the usual modes by which a soluble salt is obtained in the crystalline state?

What are the objects sought to be obtained by crystallisation?

What substances are most readily separated from one another by this process?

What circumstances influence the size of crystals?



What is the difference between water of crystallisation and water of decrepitation ?

How are crystallised salts to be dried ?

What is the difference between crystallisation and precipitation ?

What difference is there in the modes of washing precipitates and crystals ?

#### RECAPITULATION OF CRYSTALLISATION AND PRECIPITATION.

Soluble salts are usually crystallised by cooling a hot saturated solution or by evaporating the solvent. The object of the process being to obtain them free from foreign salts, and of a definite degree of hydration. Isomorphous substances are not separated by this means so readily as hetromorphous.

Crystals which are deposited slowly, and without agitation, are usually largest and most regular. Most salts crystallise in combination with water in one or several definite proportions ; those which have no water of crystallisation frequently have water mechanically held, and known as water of decrepitation.

Permanent and anhydrous salts may be dried by exposure to a warm dry atmosphere, others by the use of a bibulous material or a centrifugal drier.

The difference between crystallisation and precipitation is one of degree rather than one of kind, deposits which are but slightly soluble, and not conspicuously crystalline, being called precipitates.

Crystals are usually washed with the smallest effective quantity of cold water ; precipitates more copiously with warm water.

## LECTURE VII.

### DIFFUSION IN LIQUIDS, DIALYSIS, OSMOSIS, &c.

Diffusion—Crystalloids—Colloids—Rate of Diffusion — Dialysis — Osmosis — Hydration and Dehydration of Colloids—Infusion of Gentian, &c.—Diffusion Groups—Times of Equal Diffusion—Influence of Heat.

THOUGH the process of dialysis is not made use of in the preparation of any officinal product, the natural laws upon which that process depends are called into operation in so great a number of cases that some account of them cannot fail to be both interesting and useful.

In treating of solution, I stated that when a body is dissolved, no matter what its density may be, it ceases to be separable from the solvent by the action of gravity; and the first point to be noted now is that the dissolved bodies not only will not subside, but that, if a dense solution be placed at the bottom of a vessel, and some of the solvent be poured upon it, the dissolved matter will gradually diffuse upwards against the action of gravity till the whole liquor is uniform. And this gradual mixing cannot be looked upon as accidental, as the consequence of currents in the fluids, change of temperature, &c.; for careful experiments have shown that bodies differ in the rate at which they diffuse in this manner. A simple experiment will show this. I take two tall glass jars, one filled with pure water, the other with water coloured blue with litmus. Into the pure water is poured  $\frac{1}{2}$  oz. of caramel solution, with the aid of a long tube funnel, so that it may be carried to the bottom of the water, where it will remain as a layer of deep-coloured fluid, showing very little tendency to rise. Into the blue water is poured in the same manner

$\frac{1}{2}$  oz. hydrochloric acid, of the same density as the caramel, and mixed with gum to give it the same viscosity. The acid, however, diffuses into the blue solution at a very palpable rate, as may be seen by the change in colour which gradually takes place from the bottom upwards. In this experiment we have one of the most diffusible bodies contrasted with a body at the opposite end of the scale, and between these there is every diversity in the diffusion rates of soluble substances.

We are indebted to Professor Graham for a careful investigation of this subject. It is unnecessary for us to follow the details of his experiments, but we may with advantage note various of the results.

1st, Crystallisable substances, as a class, diffuse more rapidly than those which solidify in the gelatinous condition.

2d, Crystalline salts differ much among themselves in the rate at which they diffuse; but salts, having the same crystalline form and chemical constitution, diffuse at about the same rate.

3d, The rate of diffusion is proportionate to the strength of the solution used, and increases with a rise in temperature.

Freely diffusible bodies are called crystalloids, because of their generally being crystallisable substances; but it must not be concluded that crystalloids are all crystallisable, as hydrochloric acid, the most highly crystalloid body, and various others which also stand high in the scale, are not known in the crystalline form.

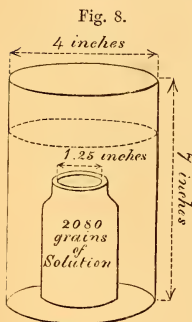
Bodies which are characterised by slowness of diffusion are called colloids, because the most perfect examples are bodies of a gelatinous nature, such as gelatine and albumen.

The following table, taken from Miller's "Elements of Chemistry," gives the comparative rate at which sundry soluble matters diffuse in water under the same circumstances:—

100 parts of water and 20 parts of	Diffused in 8 days Grains.
Chloride of sodium, . . . . .	58.6
Nitrate of soda, . . . . .	51.5
Treacle, . . . . .	32.5
Sulphate of magnesia, . . . . .	27.4
Sugar, . . . . .	26.7
Gum arabic, . . . . .	13.2
Albumen, . . . . .	3.0

These numbers, you will observe, give comparative rather than absolute results, but are equally instructive and reliable, as the same vessels, the same temperature, and strength of solution were observed throughout.

The mode of operating is as follows :—The solution to be



examined is placed in the inner jar indicated in the figure, which it completely fills; distilled water is then poured into the outer jar till it is filled up to the dotted line, every care being taken to avoid disturbing the contents of the small jar, the whole being guarded against agitation or changes of temperature during the experiment. At the end of the required time the contents of the large jar may be removed with a syphon, and evaporated to dry-

ness, together with a further portion of distilled water with which the outside of the small jar and the inside of the large one have been washed.

The weight of the residue thus obtained gives the diffusion rate of the body under experiment, compared to that of any other substance operated upon in the same way.

Vegetable tissues and animal membranes, which have the power of absorbing water freely, permit the diffusion of crystalloids to go on through their substance with very little obstruction; but they offer so much resistance to the passage of colloids that they may be considered virtually impervious to them. If a solution containing both colloid and crystalloid

matter be placed in a bladder, and the bladder immersed in water, it will shortly be found that the crystalloid matter is passing through the bladder into the water, and that the colloids will remain behind; thus a species of analysis, called dialysis, may be conducted by the different diffusibilities of the two classes of substances through the bladder. The following table, from Watt's "Dictionary of Chemistry," gives the comparative rate at which dialysis takes place with divers soluble substances:—

RATE OF DIALYSIS.

Dialyser containing 98 grams water and 2 grams of	Weight in grams diffused in 24 hours.	Dialyser containing 98 grams water and 2 grams of	Weight in grams diffused in 24 hours.
Chloride sodium, .	1·657	Extr. logwood, .	·280
Picric acid, .	1·690	Catechu, .	·265
Ammonia, .	1·404	Extr. cochineal, .	·086
Theine, .	1·166	Gallo-tannic acid, .	·050
Salicin, .	·835	Extr. of litmus, .	·033
Cane sugar, .	·783	Pure caramel, .	·009
Amygdalin, .	·517		

Results of this kind are obtained by placing the solution to be experimented upon in a dialyser consisting of a wide-mouthed bottle, the bottom of which is removed and replaced by a bladder or sheet of parchment-paper, carefully selected, free from perforations. This is immersed in distilled water, which may be replaced with fresh water at intervals, as it becomes impregnated with the crystalloid. If dialysis were allowed to go on indefinitely, the solutions on each side of the diaphragm would at last become equally impregnated with the crystalloid, and dialysis would cease; and to allow of the process going on continuously for a length of time without this occurring, a thin stratum only of the solution is put into the dialyser, and a copious supply of distilled water is placed below. The following figures (p. 76) show the usual form of experimental dialyser.

For experiment I pour into the dialyser an ounce of sarsaparilla mixture, and after the lapse of a short time test the dialysate, and readily detect the presence of iodide of potas-

sium, which has come through freely, while the dark-coloured vegetable extractive matters, which in the original mixture prevented the application of the test, have been kept back by the diaphragm.

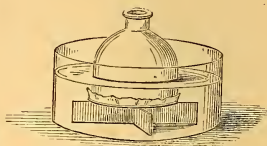
Fig. 9.



Fig. 10.



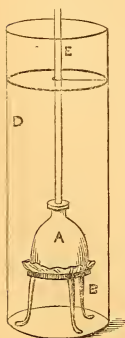
Fig. 11.



The Glass Dialyser. Support of Gutta-Percha. The whole in action.

Dialytic action has, no doubt, a very important influence upon the vital functions of animals and vegetables, and promises to reward investigators with new light on these subjects; it is not, however, in that connection that I wish to draw your attention to it, but in relation to the solution of various matters in the preparation of infusions, decoctions, &c. But before doing so, I must prepare my ground more completely by describing a third class of phenomena connected with diffusion.

Fig. 12.



If a bladder be filled with a saturated solution of carbonate of potass, while a portion of the salt is passing out, a quantity of water much in excess of the salt passes into it, so that the bladder, if full at first, may be burst by the inflow of the water.

This passage of the fluid through the bladder is called osmosis, and is distinguished as endosmosis when the water passes into the solution more rapidly than the latter passes out, and exosmosis if the outward flow preponderates.

The osmometer is an arrangement of apparatus very similar to the dialyser. The solution to be ex-

amined is placed in the vessel A, supported upon a tripod B, placed in the jar of water D. At the commencement of the experiment, the level of the water in D is made to correspond exactly with the level of the solution in the tube E. After the necessary interval, a rise in the liquid in E indicates endosmosis, or a fall indicates exosmosis. Like dialysis, the rate at which osmosis takes place can only be stated in a table indicating the results obtained in some particular course of experiments. Slight differences in the membrane would vitiate the results if different instruments were used.

In the following table, from Watt's "Dictionary of Chemistry," we have the comparison of a series of solutions, all containing 1 per cent. of solid in water, except hydrochloric acid, which contains 0.1 per cent. of H.Cl.

## OSMOSIS OF 1 PER CENT. SOLUTIONS.

Oxalic acid, . . . . .	— 148	Chloride of calcium, . . . . .	+ 20
Hydrochlor. acid (0.1 %)	— 92	zinc, . . . . .	+ 54
Chloride of gold, . . . . .	— 54	Nitrate lead, . . . . .	+ 120
platinum, . . . . .	— 30	copper, . . . . .	+ 200
magnesium, . . . . .	— 3	Chloride of copper, . . . . .	+ 351
sodium, . . . . .	+ 2	iron, . . . . .	+ 435
potassium, . . . . .	+ 18	aluminium, . . . . .	+ 540

The mark — indicates the outward flow ; the + mark indicates that the predominating flow was from the water into the solution.

Something analogous to osmosis takes place when insoluble, or imperfectly soluble colloids are exposed to the action of liquids. If you place gelatine in cold water it does not dissolve, but the water penetrates ; we cannot say into the interstices, for interstices, in the ordinary sense of the word, there are none ; but into the substance of the gelatine, we may suppose into a state of molecular combination as intimate as that of solution, this being the parallel of endosmosis. The gelatine then being placed in spirit (or some other liquids), the analogue of exosmosis will take place, a little spirit penetrating the gelatine, and a large quantity of water flowing out of it, till the gelatine becomes reduced to a state of dryness propor-



tionate to the relation which subsists between the affinity of the spirit and the gelatine for water, and the proportion of the three bodies present. This experiment, you will notice, is closely paralled to those which I used in illustration of evaporation, and the relation which subsists between the moisture of the atmosphere and of the materials exposed to it. We might almost say that the water in the gelatine had evaporated into the atmosphere of alcohol in which it has been immersed.

Our materia medica affords us numerous examples of substances which are insoluble or imperfectly soluble colloids; and our pharmaceutical operations supply equally numerous cases where these materials are exposed to the action of water, spirit, or both in succession. The subject is one well worthy of study, and, no doubt, an experimental investigation of the phenomena would yield a harvest of suggestive results.

I can at present only indicate the bearing of these phenomena upon such simple processes as the preparation of infusions.

Another force-capillary attraction, with which you are probably sufficiently familiar, also plays an important part in the passage of liquids into the substance of drugs submitted to their action; and now, supposing the ingredients for infusion of gentian to be placed in a jar and covered with hot water, capillary attraction comes into play to saturate the root with moisture, the soluble portion becomes dissolved, the cells and their contents then are subject to the action of dialysis and osmosis, the cells themselves becoming distended, and their contents being diffused more or less rapidly according to whether they are colloid or crystalloid in their nature; the mucilaginous principles being slowest in diffusing, and the alkaloids and acids more rapid.

Several of the mucilaginous principles, such as pectin and bassorin, undergo very imperfect solution; and if it is desired to extract them, the vegetables containing them must either

be thoroughly comminuted in the first place, and freely agitated during infusion, or subjected to pressure after they have been acted upon by the hot water, as their imperfect solubility and small diffusibility would otherwise prevent their satisfactory extraction. If, on the other hand, it is desired to avoid the solution of mucilaginous matters along with crystalloids, coarse comminution should be adopted, and the ingredients may be suspended in the water to avoid the necessity of stirring; cold water should be preferred, and the infusion drained off without pressure.

Turning again to Graham's results, his second induction, that isomorphous bodies have similar diffusion rates, is exemplified in the following table of diffusion groups, and it is interesting to notice that, though the time which is required for an equal diffusion of sundry compounds does not show any simple numerical relationship, if the numbers expressing time be all squared, a simple ratio at once becomes evident:—

## DIFFUSION GROUPS.

	Time of Equal Diffusion.	Ratio of Square of Time.
HCl } HI } HBr }	3.96	2
KO,HO } NH <sub>3</sub> HO }	4.95	3
Nitrate potass, ammonia, } Chloride potass, ammonia, }	7.00	6
Nitrate soda, } Chloride soda, }	8.57	9
Sulphate potass, Carbon potass, Sulphate ammonia, }	9.90	12
Sulphate soda, } Carbon soda, }	12.12	18
Sulphate of magnesia, . . .	19.80	48

The influence of heat upon the rate of diffusion is ex-

emplified in a table giving the quantity of HCl diffused at different temperatures :—

The rate of diffusion of HCl—

At	60° Fahr.	being taken at	.	.	.	1·00
	80°	„ is	.	.	.	1·35
	100°	„ „	.	.	.	1·77
	120°	„ „	.	.	.	2·18

Another table, in Watt's "Dictionary of Chemistry," gives a comparison of the time required to diffuse equal quantities of the following substances, the time required for HCl being taken as the unit :—

	Times of Equal Diffusion.
Hydrochloric acid, . . . . .	1·00
Chloride of sodium, . . . . .	2·33
Sugar, . . . . .	7·00
Sulphate of magnesia, . . . . .	7·00
Albumen, . . . . .	49·00
Caramel, . . . . .	98·00

The numbers given in one table do not exactly agree with those given in others, but I think we may set that down to comparative newness of the subject, and the tedious nature of the experiments. It is difficult to insure the perfect similarity of the results, when the circumstances are varied between one series of experiments and another, but we need not discredit the accuracy of the comparison between parts of the same table.

#### QUESTIONS FOR EXAMINATION IN LIQUID DIFFUSION AND DIALYSIS.

Define the terms crystalloid and colloid.

What effect has heat upon the rate of diffusion?

Among bodies of equal diffusibility, what other relationship is frequently observed?

What is meant by dialysis?

Define the terms osmosis, endosmosis, and exosmosis.

## RECAPITULATION.

Various bodies in solution in water diffuse into pure water at very unequal rates. Those which diffuse most rapidly are called crystalloids, those which diffuse slowly are called colloids. Heat increases the rate of diffusion. Isomorphous compounds have usually about the same diffusion rate. Hydrated membranes afford little obstruction to the passage of crystalloids, but are almost impervious to colloids. These classes of bodies being separated by this means, the process is called dialysis. If a hydrated membrane has water on one side and a solution on the other, they flow through the membrane at different rates; this flow is called osmosis. If the flow of water into the solution preponderates, it is called endosmosis; if the flow of the solution into the water preponderates, it is called exosmosis.

## LECTURE VIII.

### EVAPORATION, BOILING, FUSION, AND CALCINATION.

Vegetable Juices—Evaporation over Naked Fire—Mechanical Stirrers—Sand Bath, Water Bath, Saline Bath, &c.—Evaporation *in Vacuo*—Rate of Evaporation and of Vaporisation—Fusion—Behaviour of Crystalloids and Colloids at their Melting Points—Latent Heat of Fluidity—Behaviour of Fusible Crystalloids and Colloids under the Action of Heat and Solvents—Utensils for Fusions—Calcination.

IN treating of the desiccation of herbs, I pointed out the general laws of evaporation, and described some appliances which are available in the evaporation of water. We have now to consider such appliances to be used, and such general rules to be observed, in the evaporation of liquids, as did not then come under our notice.

Vegetable solutions for the preparation of extracts being more prone to decomposition than the herbs from which they have been extracted, require considerable care and great promptitude in their reduction to such a degree of concentration as puts them out of danger of fermentation; compared to the drying of herbs, we have now a much greater quantity of water to evaporate; the active principles are deprived of the protection which their natural covering generally affords, and the liquid, instead of being diffused, as it is in the leaves and branches, allowing free circulation for the air, now lies compactly at the bottom of the pan; there is consequently tenfold urgency to have recourse to every artificial means of expediting the process, and to have a vigilant eye upon all the chances of injury to which the liquid is exposed.

With the view of expediting evaporation, the pans used for

this purpose are constructed very wide in proportion to their depth, and when set upon the fire for active ebullition, which is sometimes admissible, only the centre of the bottom of the pan is exposed to the direct action of the fire; the residue, which is apt to adhere to the pan just above the surface of the boiling liquor, is thus in a measure protected from scorching; but as a further protection, it should be frequently washed down from the sides by stirring the hot liquor, and driving it from one side to another. Evaporation over a naked fire will require pretty constant attention in all cases where an organic material is being operated upon.

In most cases, at a certain stage in the process, a film appears upon the surface of the liquid which very much retards its evaporation. This is to be avoided by constant stirring, which is usually performed by hand, except in laboratories where evaporation is so constantly going on as to make it worth while having mechanical contrivances to save the time of the operator. A circular movement may be given to the stirring-rod by attaching it to a wheel moved by machinery, but as this moves in one direction at a constant rate it is not nearly so effective as stirring by the hand, which moves the fluid in all directions, and most effectually breaks the pellicle. A better arrangement for mechanical stirring is to have the stirrer hinged above the pan, and moved backward and forward, the bottom of the stirrer being like a broad spade almost fitting the bottom of the pan, and the hinge being in the focus of the pan, or rather in the centre of the imaginary sphere of which the pan is a segment; the blade of the stirrer having larger perforations or notches through which the liquor will flow while the stirrer moves from side to side.

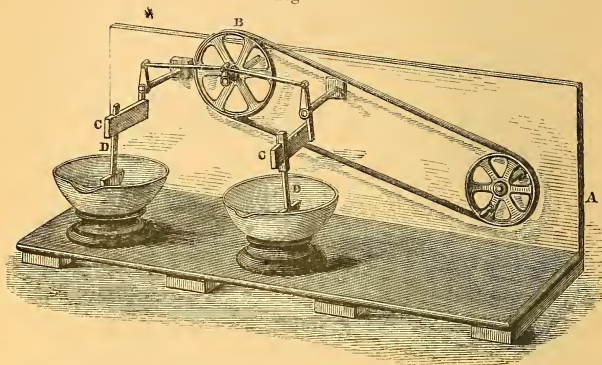
The cut exhibits Mr Reynolds's system of stirring, as described at the Pharmaceutical Conference, 1867. His description will be found in "Pharmaceutical Journal" 1866-7, page 221.

The material of the pan should be copper, block tin, or enamelled iron; the first and last being preferred for

movable pans, the second for pans fixed in steam jackets, &c.

Various arrangements are made for regulating the heat as applied to evaporating, boiling, or distilling apparatus.

Fig. 13.



Reynolds' Mechanical Stirrer.

Appliances of this kind are called baths, and have distinctive prefixes, according to the material used to regulate the conveyance of heat from the fire to the fluid under operation.

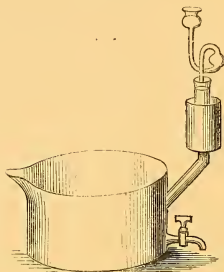
Sand, water, steam, oil, glycerine, saline solution, and fusible metals are all in turn used for this purpose, each having advantages for particular operations.

The sand bath consists simply of sand laid upon an iron plate heated by the fire. It prevents rapid changes of temperature, but does not afford security against too great heat. The saline bath may be used when a temperature a few degrees above boiling water is required, and where a heat much higher would be injurious. It consists of a pan set in another pan, charged with a saturated solution of some salt, which boils at about the temperature within which it is desirable to keep the contents of the bath. As long as any water remains in the lower pan, the temperature never rises above



the desired point ; and as long as the water is replenished in quantities insufficient to dissolve the whole of the salt, the temperature will never fall below the required point. The cut represents a bath of this kind,

Fig. 14.



Saline Bath.

The same arrangement of pans without salt constitutes the ordinary water bath in which the lower pan is always retained at  $212^{\circ}$  Fahr. ( $= 100^{\circ}$  C.), the upper, of course, being a few degrees less ; thus the saline bath may be used for boiling down watery liquids, while the water bath can only be used for evaporating them below the boiling-point.

The steam bath admits of a certain range of temperature from the boiling point upwards, according to the pressure which the apparatus is calculated to sustain. There are more difficulties in getting all the joints sound in proportion as the pressure increases, but in any considerable laboratory this is amply compensated for by the convenience of having one boiler and fire supplying heat to almost any number of pans, the pans being connected or disconnected at any moment by simply turning the steam-cock with which each pan is supplied.

The steam bath is constructed of two pans rivetted or bolted together at the edges, the lower pan being deeper than the upper ; there is a space between, into which the steam is allowed to enter, a provision being also made to carry off the water resulting from condensation.

The evaporation of a vegetable liquor cannot safely be conducted over the open fire after it has acquired the consistence of syrup, but the great majority may be evaporated in a steam bath, working with 4 or 5 lbs. pressure, without any fear of injury; the Pharmacopœia, however, directs the evaporation of many of the extracts at a temperature not exceeding  $140^{\circ}$  or  $160^{\circ}$  Fahr., at other times directing simply that a water bath should be used, in which case the heat of the extract will be about  $170^{\circ}$ ,  $180^{\circ}$ , or  $190^{\circ}$  Fahr. so long as it remains fluid, but may rise to within a few degrees of the boiling point as it becomes thick, and evaporation goes on so much more slowly. In any case where it is imperative to keep the temperature below  $140^{\circ}$  to  $160^{\circ}$  Fahr., the use of the thermometer becomes necessary. It may either be placed in the extract or in the water of the lower pan, which is in some respects more convenient; but in this case it must be remembered that the extract will be a few degrees, though an uncertain number of degrees lower than the thermometer indicates.

Below the boiling point the rate of evaporation is increased the more nearly the temperature approaches  $212^{\circ}$  Fahr.; the larger the surface exposed to the air, the more perfectly the surface is kept free from pellicle, and the more rapidly the air passes over the surface,

At the boiling point the rate of evaporation (or vaporisation, as it is then called for distinction) increases in proportion to the supply of heat to the pan, and the amount of pan surface which is covered by the liquor.

So far, I have directed your attention to the evaporation of liquors under ordinary atmospheric pressure. A most important improvement in the process is to conduct it in a closed vessel, constructed like the steam bath, with the addition of an air-tight cover, the air being then exhausted by means of an air-pump worked by a steam-engine. The evaporation, under these circumstances, takes place with equal velocity at  $80^{\circ}$  to  $90^{\circ}$  Fahr. lower temperature. Rapid evaporation can thus be accomplished without fear of injury from over-heating,

and contact with air is entirely avoided. Extracts prepared in this way generally preserve in a higher degree the natural flavour and odour of the plant.

The evaporation of inorganic solutions requires usually much less care, brisk ebullition being generally admissible ; they do not call for any particular comments at present.

When ebullition is performed for the sake of the decoction, several points will be advantageously the reverse of those which have just been commended, as in this case any considerable evaporation is to be avoided.

The pan should be deep and covered, and as soon as boiling commences, it may be removed to the side of the fire, where the heat is just sufficient to keep it gently boiling for the required time. Common coffee-pots are sometimes used, but are objectionable, as being difficult to clean thoroughly after each operation.

#### FUSION OR LIQUEFACTION BY HEAT.

The term fusion is commonly restricted to the liquefaction of such substances as require for this purpose a high temperature, but it is both more convenient and more philosophical to treat all together ; the melting of ice, or of cast iron, being just the same as regards the principles involved, and the appliances to be used for the one are linked in an unbroken chain of slight modifications with the arrangements required for the other.

The pharmaceutical operations in which fusion is required are very numerous, but, generally speaking, also very simple ; they consist of such processes as making plasters or ointments, the casting of caustic, nitre balls, &c., the purification of bismuth, sulphuret of antimony, gum resins, &c.

Fusible solids, when gradually heated to their melting point, do not all behave in the same manner ; thus wrought iron becomes soft, plastic, and adhesive before it melts, but cast iron becomes brittle and crumbly ; other substances, such as nitrate of silver, or of potassium, undergo no very visible

change until liquefaction takes place. It has been supposed that bodies change from the crystalloid to the colloid state of matter as they assume the plastic state which precedes fusion. When fusion commences, they combine with heat in such an intimate manner, that the addition of heat does not cause any rise in temperature till the whole of the substance be liquefied; the heat thus disappearing is called latent heat of fluidity, its quantity varying very much with different materials, as is indicated in a few examples in the following table:—

## LATENT HEAT OF FLUIDITY.

	Fahr.		Fahr.
Water, . . .	142°	Bismuth, . . .	22°
Nit. soda, . . .	113°	Sulphur, . . .	16°
Nit. potass, . . .	85°	Lead, . . .	9°
Zinc, . . .	50°	Phosphorus, . . .	9°
Tin, . . .	25°	Mercury, . . .	5°

Thus 1 lb of ice at 32° Fahr. will absorb as much heat in becoming water at 32° Fahr., as would have sufficed to raise 1 lb of water at 32° Fahr. up to 174° Fahr. Or, to put it in another way, 1 lb of ice at 32° Fahr., and 1 lb of water at 174° Fahr., when mixed together, will make 2 lb of water at 32° Fahr.

This law has a practical bearing upon the melting of various substances, which are liable to be injured by exposure to a heat a little above their melting point; thus, a pan of ointment or plaster may be kept upon the fire with little fear of injury so long as a portion remains unmelted, but to keep it there, even a few minutes, after fusion is complete, would be at considerable risk of its injury, for then the heat added causes a rapid rise of temperature.

It may be stated as a rule, that fusions should be effected at the lowest practicable temperature. The next table shows the melting points of various substances in which pharmacutists are interested:—

## MELTING AND FREEZING POINTS.\*

	Fahr.		Fahr.
Mercury,	— 39°	Spermaceti,	+ 112°
Oil vitriol,	— 30°	Wax,	+ 143°
Proof spirit,	— 7°	Iodine,	+ 224°
Bromine,	— 4°	Sulphur,	+ 239°
Oil turpentine,	+ 16°	Camphor,	+ 303°
bergamot,	+ 23°	Tin,	+ 451°
cinnamon,	+ 23°	Bismuth,	+ 512°
Water,	+ 32°	Nitrate potass,	+ 642°
Oil aniseed, about	+ 64°	Lead,	+ 620°
Lard, about	+ 90°	Zinc,	+ 773°
Phosphorus,	+ 111°		

A piece of wax may remain immersed in cold olive oil for a long time with little appearance of change, but if the wax be melted, solution immediately takes place, and as it cools the wax continues in a condition of imperfect union with the oil; if a piece of spermaceti be treated in the same way, it is seen to crystallise as the oil cools; and by pressure, it may be in great measure separated again from the oil, the solution of the wax and the spermaceti being equally perfect at their melting points, but the wax remaining in more intimate union on the subsequent cooling.

If the experiment be repeated with rosin, a thick semifluid is obtained on cooling without any separation of the rosin. In these three cases we have the rosin a colloid, the spermaceti a crystalloid, and the wax an intermediate body, and they illustrate the different behaviour of fusible bodies belonging to these classes when subject to heat, the spermaceti undergoing very little softening previous to fusion, while the rosin passes by imperceptible degrees from the solid to the liquid state.

In the production of ointments and plasters, that condition between hardness and fluidity is aimed at, which is best obtained by the presence of at least one body of a colloid nature. I must state, however, that I am at present using the terms colloid and crystalloid without reference to their

\* From Redwood, Miller, and Gmelin.

diffusive powers, as this property has not yet been investigated, for many substances insoluble in water.

The appliances used for fusions are very various, depending upon the chemical relationships and the melting point of the substance in hand. Thus a water bath of glazed earthenware is very suitable for ointments and most things requiring a heat below  $212^{\circ}$  Fahr. Copper pans are suitable in many cases where a slightly higher heat is required, or where a material, from its hardness or adhesiveness, would endanger earthenware in its subsequent removal. Copper pans, however, are quite unsuitable for melting sulphur, nitrate of potass, hydrate of potass, or nitrate of silver, as these materials have a powerful action upon copper; iron is much less affected by them. Platina vessels may be used for the fusion of nitrate of silver, &c., but would be destroyed by sulphur. Porcelain may be used for sulphur or nitrates, but would be attacked by caustic alkalies. Fire-clay crucibles are little acted upon by most pharmaceutical products, except the caustic alkalies, but are too porous to be suitable for valuable materials like nitrate of silver, they are specially suited to fusion of metals, and operations requiring a very high temperature.

They are subject to two inconveniences, viz., that they are liable to crack and will rarely bear more than two or three heatings, unless used with considerable care, and only exposed to a moderate heat. The absorbent character of the material, also, will commonly render a crucible which has been used for one operation unfit for another of a different nature. I have frequently used crucibles of fire-clay mixed with plumbago, and find them capable of standing a great many re-heatings without cracking, but they become rather crumbly and very porous with long use, as the plumbago burns out.

Calcination is rarely performed by the pharmacist; the inconvenience of having many different small operations, and the cumbrous appliances required for their performance, in great measure excludes calcination from the list of laboratory processes, such chemicals as require it being better made on the

large scale, where the apparatus is in constant use for the same purpose.

The process consists essentially in exposing a substance to a high heat, generally with the view of driving off some volatile matter (though it may be sometimes with the view of absorbing oxygen), the product being in a powdery or friable condition, and not fused. The matter to be calcined may be placed in a crucible, and exposed to the heat of an ordinary furnace, or a reverberatory, until the required change has taken place. The time required is shorter as the quantity operated on diminishes, and as the heat of the furnace increases. It also depends in some measure upon the nature of the atmosphere with which the material is surrounded, the action being in great measure comparable to that of the drying closet. As it is difficult to evaporate water in an atmosphere charged with steam, so it is difficult to drive off carbonic acid from carbonate of lime or magnesia in an atmosphere of carbonic acid, the evolution of the gas taking place much more readily in a current of air, especially if accompanied with carbon and hydrogen, which decompose the carbonic acid into carbonic oxide at the moment of its liberation. Thus, limestone is much more easily converted into quicklime, if burnt in contact with the fuel, than if heated in a close retort. Artificially prepared carbonate of lime is more easily reduced to the caustic state than the native lime stones, and if pure lime be required, the precipitated carbonate may be calcined in a covered crucible at a heat below whiteness. Magnesia and oxide of zinc, prepared from the carbonates, require a low red heat. Burnt alum and dry sulphate of iron are produced by a heat considerably below redness, a higher heat driving off not only the moisture but the acid. Sulphate of manganese is purified from iron by calcining at a full red heat, which does not decompose the sulphate of manganese, but converts the sulphate of iron into sulphurous acid, which is driven off, and peroxide of iron, which is afterwards separated by dissolving the manganese salt, and



filtering out the insoluble peroxide. In calcining the sulphate of iron for use as dried sulphate, the danger is in having the heat too high; in calcining the sulphate of manganese to purify it from iron, the danger is of having the heat not high enough.

### QUESTIONS FOR EXAMINATION.

What temperatures are usually desirable in the evaporation of vegetable juices?

What are the disadvantages of temperatures, too high or too low?

What are the usual modes of regulating the temperature of evaporating liquors?

What is the essential advantage of a water bath over that of a sand bath or naked fire?

How does stirring promote evaporation?

What circumstances expedite the evaporation of a liquor kept below its boiling point?

What circumstances determine the rate of vaporisation of a liquor at its boiling point?

In what respect should we vary the appliances for boiling when our object is to produce a decoction?

What difference is observed in the behaviour of crystalloid and colloid bodies just below their melting points?

What useful application have we for fusible colloids in pharmacy?

What is meant by latent heat of fluidity? Is it of variable amount?

What is the nature of calcination?

### RECAPITULATION.

Evaporation of vegetable juices requires to be performed promptly to avoid decomposition. They are liable to injury by too high a heat, or too long a continuance of a temperature insufficiently high; the temperature is conveniently regulated

by water baths, steam baths, &c., but is not safely regulated by a sand bath.

Constant stirring is in all cases advantageous, and most important in evaporation by means of sand bath or naked fire.

The rate of evaporation below boiling point increases the more nearly that point is approached, the larger the surface exposed to the air, the more rapid the current of air, &c.; at the boiling point the rate of vaporisation depends mainly upon the intensity of the heat to which the pan is exposed, and the extent of surface exposed to it. In making decoctions, the vessel should be deep, narrow, and covered, to avoid loss by evaporation.

Fusible colloids become plastic a little below their melting points. Fusible crystalloids sometimes become more brittle before they melt.

During fusion a certain amount of heat disappears; this is known as the latent heat of fluidity, and varies much with different bodies.

Fusible colloids are required in plasters and ointments, to give them adhesive and plastic qualities.

Calcination consists of heating a substance strongly, to drive off volatilisable constituents, or cause oxidation, and leave a powdery residue.

## LECTURE IX.

### DISTILLATION AND SUBLIMATION.

Forms of Apparatus—Alembic—Retort and Receiver—Flask and Safety Funnel—Tubulated Condenser and Receiving Bottle—Liebig's Condenser—Boiler and Worm Tub—Fractional Distillation—Boiling Points of Mixed Liquids—Boiling Points of Essential Oils—Distillation of Essential Oils—Condensing Power of Cold Water—Mitscherlich's Condenser—Bumping—Sublimation—Calomel Hydro-sublimation and Dry Sublimation—Iodine.

WE have considered the conversion of water and other liquids into the gaseous state, and have now to study the means of reconverting their vapours into liquids. The two processes taken together constitute distillation. The objects of distillation may be either the separation of two mixed or combined substances or the combination of elements into new compounds, and occasionally both combination and separation are effected at once.

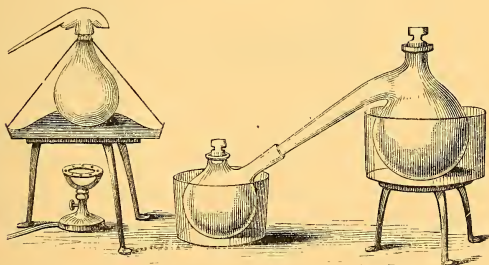
There are many interesting facts connected with distillation to which I shall allude, after briefly describing various kinds of distilling apparatus, noting the kind of operation for which they are suitable, and the points to be attended to in each.

The first is the old alembic, a piece of apparatus rarely now put to practical use. It consists of a flask fitted with a head, in the form of two concaves, the upper being the condensing surface, the lower forming a gutter to collect the condensed fluid, which is subsequently conveyed away by the tubulure; it may be used for distilling small quantities of corrosive liquids, which are easily condensed. It has the advantage of being entirely of glass, there being no cork or india-rubber connections and no luting, but the disadvantage of being very

inefficient in condensing power. The body, or flask-like portion, with its contents, should be set in a sand bath, a cone of paper or card placed over it so as to keep the whole flask hot, and then the head put on; the cone in some measure prevents the heat reaching the head, but for its more effectual cooling it should be fitted with a cap of thick cloth or filtering paper kept constantly moist. The retort and receiver is a much more convenient and useful arrangement, and now that they can be had with the beak of the retort ground into the receiver, forming an air-tight joint, there is scarcely any purpose to which the alembic may be put that is not better accomplished by this apparatus. The receiver may be immersed in cold water, while the retort is heated by a water bath, sand bath, gas furnace, or any other means. The two preceding arrangements are scarcely suited to anything beyond experimental purposes; for operations on a larger scale more substantial and capacious vessels must be used, which are not easily fitted air-tight by grinding.

The following is an arrangement which I have frequently adopted for working quantities from a pint up to half a gallon:—

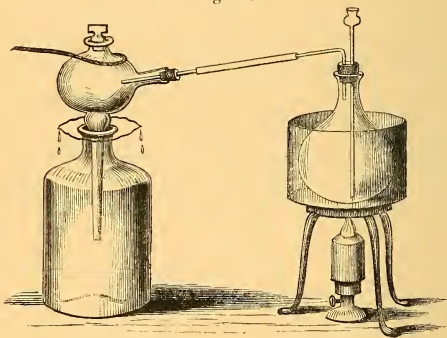
Fig. 15.



A large flask is fitted by means of a cork with a tube funnel and bent tube; the flask is set in a water bath over a gas furnace. The bent tube is connected by means of an india-rubber tube to a second glass tube, fitted by a perforated cork to the

tubulated receiver (which, when thus used, would be more properly termed the condenser); round the top of the tubulure of the receiver is fitted a collar of leather or sheet india-rubber, and then the tubulure is inserted in a half-gallon bottle. Round the top of the receiver is carried a small india-rubber tube supplied with water, and perforated so that the water shall trickle down in small streams, and over this a loose calico cover to promote the spread of the water over the surface of the condenser. As it runs down, the collar fitting tightly round the tubulure, the water is carried safely over the mouth of the bottle; it then runs down the sides of the bottle, keeping it also cool. All parts of the apparatus are air-tight, except

Fig. 16.



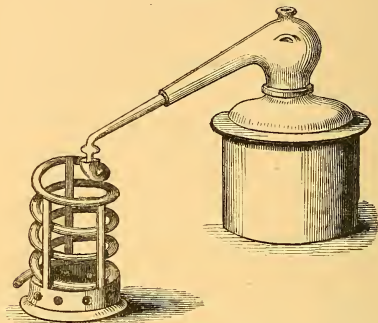
the fitting of the condenser into the bottle. Any vapour escaping condensation in the condenser passes into the bottle, where condensation is completed. If any sudden burst of vapour takes place, the loose fitting of the condenser to the bottle affords the means of escape, without which the bursting of the flask, or ejection of its contents through the funnel, would be endangered; it also affords means of admitting air in event of there being excessive condensation. The tube funnel attached to the flask answers the double purpose of safety against sudden condensation, and a means of refilling the flask when the

great bulk of the liquor has distilled. If the condensed liquor rises in the bottle above the bottom of the tubulure, and sudden condensation takes place, the contents of the bottle would be drawn up into the receiver, and possibly even into the flask, were it not provided with the tube-funnel. This arrangement is suited for recovering the spirit in the preparation of alcoholic extracts, &c. It may also be used for the distillation of spirit of nitre, &c. In which case, or in any others where the india-rubber tube is objectionable, the bent tube from the flask may be fitted to the condenser direct; but this makes the apparatus rigid, and consequently less convenient. Liebig's condenser is an efficient and convenient piece of apparatus suited for processes of any moderate magnitude. It consists of two tubes fitted one within the other, with a space between for water. The inner tube is of glass, the outer of glass, metal, or any other suitable material; they are placed either vertically or in a sloping position, the vapour to be condensed being passed *down* the inner tube, while cold water is made to flow *up* the space between the two. This is economical of the water used for condensing, as each portion of water in its passage up the tube continues to abstract heat from the vapour, till at last it may flow away at almost as high a heat as the vapour enters; and the vapour as it descends parts with heat at every step, always coming into juxtaposition with water colder than itself till it is condensed into a liquid, and flows out at the bottom, it may be not much warmer than the cold water supplied for its condensation. One great recommendation to Liebig's condenser is, that when sufficiently large it is thoroughly efficient, at the same time that it is easily cleaned, and presents no junctions liable to be acted on by corrosive chemicals; it may be used in turn for aromatic waters or spirits, for ether, nitric acid, or bromine.

Stills of a few gallons' capacity are often made of stoneware, in the form of a retort, or more advantageously, the body and head may be made separate, the junction being made air-tight by grinding the two parts together or by luting. They are suitable for the distillation of acids and

other corrosive materials, or such as require a high heat. In this case they are set in sand, contained in an iron pot, heated by a furnace. If used for distilling oil of vitriol, a hood should be placed over the still head to protect it from cold draughts, which are liable to cause fracture even in stone-ware when heated to the boiling point of the strong acid.

Fig. 17.



Doulton and Watts' Retort, with head, dip-arm, and worm.

When distillation is performed with the view of separating two or more liquids having different boiling points, it is usual to supply the boiler with a thermometer, and to collect in different vessels the liquors condensed during the time the temperature continues between certain given points. This process is called fractional distillation, and is largely used in separating the coal-tar oils and petroleums into their more and less volatile constituents. A similar object is sometimes effected by passing mixed vapours through a series of condensers, the temperatures of which are regulated, and each one being cooler than that which precedes it. This method is sometimes used in the distillation of spirit, to save a second rectification.

By way of a further illustration of this method, we will suppose it is desired to separate water and alcohol from ether, in which case the condensers may consist of three Woulff's bottles. The vapours from the still being passed into the



first, which is placed in a tub of water at  $190^{\circ}$ , water vapour will here be condensed; but its temperature being above the boiling point of alcohol, its vapour will pass on to the second, the temperature of which is regulated to  $120^{\circ}$ . Here the alcohol condenses, its boiling point being  $170^{\circ}$ ; but the ether, the boiling point of which is  $98^{\circ}$ , passes on to the third bottle, which should be kept at the lowest convenient temperature. It does not often happen that there is occasion to separate these three from one another, but the separation of water from alcohol is done in this way to save a second distillation. The last bottle may be replaced by any other form of condenser; and if there are only two vapours to condense, only one bottle need be interposed between the still and the ultimate condenser. On the other hand, if coal oils are being distilled, any additional number of bottles may be used, according to the number of qualities into which it is desired to divide the distillate. This method is known as fractional condensation. In theory we might expect that the separation of mixed fluids of different boiling points would be perfectly effected in this way; but this is not the case, though water will not boil below  $212^{\circ}$  at ordinary pressure, yet it will evaporate rapidly at  $190^{\circ}$ ; consequently a considerable portion of its vapour may be carried along with the alcohol, and condensed in the next receiver. This is a species of action constantly going on in the still. The contents of the still being two liquids of different boiling points which are freely intersoluble, the boiling point is usually found somewhere between the boiling points of the two liquids; but if they are not freely intersoluble, the mixture boils at about the boiling point of the more volatile, and yet the less volatile will distil to a large extent along with it. The former case is illustrated in the distillation of spirit which boils between the boiling points of alcohol and water, the boiling point varying according to the proportion of the two; and as the distillation proceeds, the more volatile liquid coming over most rapidly, the boiling point of the mixed liquor in the still is continually rising, till

the whole of the alcohol has come over, and the temperature of 212° Fahr. has been attained.

The latter case is illustrated in the distillation of essential oils. The oils boil at a much higher temperature than the water; yet a mixture of the two boils at nearly the same heat as pure water, and the boiling point does not rise so long as water remains in the still; but if an essential oil be distilled without the presence of water, the case is very different. Most of the essential oils, as obtained by distillation from plants, contain two or more substances of different degrees of volatility, which, though not freely soluble in water, are freely soluble in one another, and when subjected to dry distillation, the more volatile coming over first, the boiling point continues to rise till a uniform substance remains in the still, or till the whole has been brought over. The table shows the boiling points of sundry essential oils; but though these heats have been indicated as *the* boiling point, it must be remembered that the point is, in many cases, very variable. For example, the oil obtained from the distillation of hops with water will boil at 284° Fahr.; but it be kept boiling for a while, the temperature may rise as high as 600° Fahr. It is not unlikely that climate, season, and soil may influence in a considerable degree the proportions of the sundry volatile matters contained in plants, and consequently influence the boiling points of their essential oils. This seems the more probable when we observe the differences of the boiling points as determined by various authorities.

#### BOILING POINTS OF VARIOUS ESSENTIAL OILS.

	Fahr.		Fahr.
Hops, from . . .	284°	Chamomile, . . .	347°
to . . .	600°	Bergamot, . . .	361°
Mustard, . . .	298°	Aniseed, solid, . . .	432°
Turpentine, . . .	520°	Caraway, . . .	437°
Juniper, . . .	320°	Cloves, . . .	469°
Savine, . . .	320°	Ginger, . . .	475°
Thyme, . . .	329°	Cubeb, . . .	500°
Lemon, . . .	343°	Copaiba, . . .	500°*

\* Compiled from Miller's "Elements of Chemistry."

Great care is requisite in the distillation of essential oils to avoid empyreuma. The great bulk of the material to be operated upon, its liability to scorch upon the bottom of the still, and thus injure both the still and its contents, is a source of constant trouble, unless special provision is made to obviate these accidents. The accompanying table shows the highest and lowest estimate of the yield of essential oil from 100 lbs. of various vegetable materials, the number indicating ounces and decimal points. Thus, 100 lbs. of dry chamomile flowers yield 1·38 ounce essential oil; another sample of the same yielded 5·33 ounces; 100 lbs. of good mace yielded 154·0 ounces of essential oil, and 100 lbs. of worm-eaten mace only yielded 65·60 ounces.

100 lb. of		Essential Oil in ounces.
Bitter almonds,	yield . . .	0·38 to 7·70
Chamomile flowers, dry,	„ . . .	1·38 „ 5·33
Caraways, . . .	„ . . .	46·00 „ 70·00
Cloves, . . .	„ . . .	112·00 „ 272·00
Juniper berries, . . .	„ . . .	7·75 „ 16·15
Lavender herb, . . .	„ . . .	9·00 „ 15·00
Rose flowers, . . .	„ . . .	0·25 „ ...
Black mustard-seed, . . .	„ . . .	3·90 „ 9·10
Peppermint, fresh, . . .	„ . . .	3·40 „ 6·25
dry, . . .	„ . . .	15·62 „ 21·00
Nutmegs, . . .	„ . . .	64·10 „ 108·25
Mace, . . .	„ . . .	65·60 „ 154·00 *

To avoid the burning, the materials are sometimes placed in a chamber or cucurbit, and only exposed to the current of steam which passes through them on its way from the boiler to the condenser; others put the herb into the water, but keep it off the bottom of the boiler by means of a false bottom of wickerwork or perforated metal.

It is found that the oil comes over more freely if the material is in actual contact with the water, than if it is only exposed to the action of the steam; consequently, if an aromatic water is desired, the latter process may be used; but if the object be to separate the oil, direct contact with the water is

\* From Christison's "Dispensatory."

to be preferred. The undissolved portion of oil may be separated, and the water returned to the still with more of the herb, by which means the loss of oil from its solubility in water is in great measure avoided.

The quantity of cold water required to condense the products of distillation is very considerable, and while it is necessary to make ample provision, it is also necessary to have a regard to its economy by using efficient condensers, &c. The ordinary worm-tub is very efficient, easily constructed, and not liable to get out of order; but has one great disadvantage in the difficulty of getting it thoroughly clean after it has become impregnated with essential oil, &c. It is, therefore, better adapted to the use of a manufacturer, who has to prepare large quantities of a small number of preparations, than for the ordinary pharmacist who has many small processes to conduct. It consists of a pipe of copper, block-tin, stoneware, or other material, coiled round the inside of a tub, the vapours passing into the pipe at the top, and the condensed liquor running out at the lower end, which passes through the side of the tub just above the bottom. A constant stream of cold water is kept running into the tub at the bottom. As this becomes heated and rises to the top, it escapes by an orifice provided for that purpose. The efficiency of a condenser depends upon the lowness of the temperature of the condensing pipe at its termination: the economy of the arrangement depends upon no water escaping till it has become as nearly as possible the heat of the vapour entering the condenser. By supplying the cold water to the bottom of the worm-tub both these points are gained.

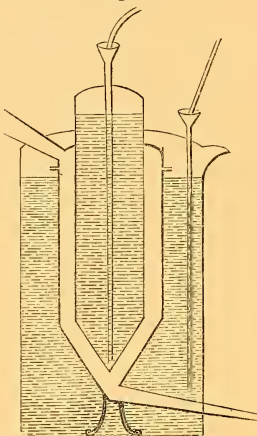
We may state the latent heat of water roughly as 1000° Fahr. (= 555° C.) The available absorbing power of water is the difference between the temperature at which it is supplied, say 60° Fahr. (or 15° C.), and that at which it afterwards runs to waste, say 160° Fahr. (70° C.), in which case its absorbing power would be 100° Fahr. (55° C.), and it would be necessary to supply 10 gallons of cold water for the con-

densation of one gallon of aqueous distillate. If the waste water flows away at a higher heat, of course so much less may suffice; but, on the other hand, if the condensed liquor flows off at a temperature below  $212^{\circ}$  Fabr. ( $100^{\circ}$  C.), so much more must be allowed for this absorption.

The condenser of Mitscherlich is also a convenient and efficient piece of apparatus, which is easily cleaned. It consists of a condensing chamber

Fig. 18.

made of metal, tin being the material most generally suitable; inside this chamber is a cylinder kept supplied with cold water, which runs in at the bottom and flows over the top into the tub in which the condenser is placed. The tub is also supplied with cold water in the same way. The chamber being made in two pieces fitted together by flanged rims, it can be opened, and the essential oil, &c., thoroughly cleaned off between one operation and the next.



Mitscherlich's Condenser (Redwood).

In many processes of distillation, or even of boiling in an open glass vessel, the phenomenon known as bumping is a constant source of annoyance and even danger. The phenomenon has not yet been satisfactorily explained, though it has received the attention of various illustrious men; it is found that many liquids will boil freely and quietly at first, but after a while the ebullition takes place in bursts sometimes so violent as to eject the whole contents of the flask or retort, or even to break the glass by the violence of the concussion. The temperature of the liquid is found to rise much above its usual boiling point between each burst of vapour, the accumulated heat then suddenly evolves a large bulk of

steam, and the temperature falls at once to the normal boiling point.

This phenomenon is most noticed when boiling liquids in glass vessels with smooth clean surfaces. Indeed, Mr Tomlinson has shown that pure water cannot be boiled in a perfectly clean glass without considerable bumping. It has been a common practice to put a few chippings of platinum foil, or a few chips of flint, into retorts with the view of facilitating the steady evolution of vapour; but it is found that after a time these nuclei cease to have the effect, and bumping recommences. Mr Tomlinson's remedy, which he says is perfectly successful, is to put a few fragments of charcoal into the retort; and where charcoal is not admissible, as in the distillation of oil of vitriol, he recommends pumice-stone, the efficacy of these substances no doubt depending upon the singular and unexplained relationships which subsist between some porous solids and gases. Spongy platinum would probably prove useful for the same purpose.

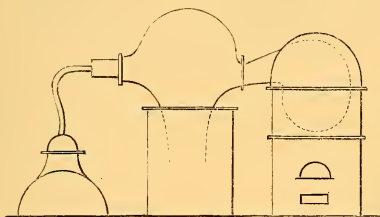
Sublimation is closely allied to distillation in its nature, but requires sundry modifications in the apparatus, in consequence of the vapours being more readily condensed, and the necessity for the condensing chamber being sufficiently open to permit the ready removal of a solid material. It is a process not often conducted on the small scale, the principal pharmaceutical products in the manufacture of which it is used are sulphur, ammoniacal salts, mercurial salts, iodine, arsenious acid, benzoic acid, and camphor. The mode of condensing the product depends upon the condition in which it is desired to obtain it; thus camphor is preferred in the massive state, calomel in an impalpable powder, and sulphur in both forms.

When it is desired to condense the sublimate in compact masses, the atmosphere of the condensing chamber should be so hot that the vapour does not condense till it comes in contact with the sides of the chamber; it will then deposit upon them layer after layer, in a condition more or less crystalline and compact.

When the pulverulent form is required, the vapours should condense from coming in contact with a cool atmosphere, which may be managed by having the condensing chamber large and sufficiently ventilated. Attention must also be paid to the degree of volatility of the material in hand; thus iodine and camphor volatilise freely at atmospheric temperatures, and a current of air passing through the condensing chamber would entail considerable loss, while calomel and sulphur do not evaporate without a considerable rise of temperature. Ventilation is out of the question in subliming sulphur, as it inflames below its boiling point; but with calomel, ventilation either with air or steam is the usual process.

The following figures may be taken as representing the process of sublimation in a sufficient variety of forms for our present purpose. First, the hydrosublimation, as contrived by

Fig. 19.



Hydro-sublimation of Calomel ; Howard & Co.'s process, as contrived by Mr Sewell.

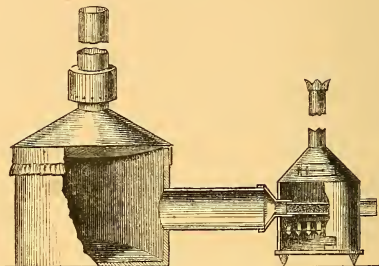
Mr Sewell of Howard & Co. The crude calomel being introduced into the stoneware retort heated over a furnace, the vapours are conducted into a stoneware receiver which has three openings, the first to receive the calomel vapour, the second to receive a jet of steam, and the third dipping into water carries down the calomel and condensed water together. The calomel is thus obtained in an impalpable powder, and any corrosive sublimate which may be condensed at the same time is washed away in the water.

The other method is to heat the crude calomel in a stone-



ware tube, through which a gentle current of air is constantly flowing into a chamber of such size that it never approaches the heat required to maintain calomel in the gaseous state. It thus condenses by contact with the comparatively cool air of the chamber, in a condition so fine as to resemble smoke, and would thus pass away through the ventilating flue of the chamber, unless ample space were allowed it for subsidence,

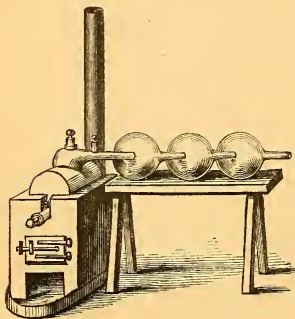
Fig. 20.



Dry Sublimation of Calomel (Redwood).

or the air filtered through a cloth as it leaves the chamber. Whichever method be adopted, in the case of calomel it is

Fig. 21.



Lead Still; glass condensers (Parcira).

always necessary that washing should follow the sublimation, not only because the crude calomel always contains some portion of perchloride of mercury which is sublimed at the same time, but even pure washed calomel, if subjected to a second sublimation, is, to a very small extent, decomposed into corrosive sublimate and metallic mercury.

The third figure represents the sublimation of iodine. The crude alkaline iodides are put

into the lead still with oil of vitriol and black oxide of manganese; on the application of heat the iodine vapours pass over, and are condensed in the series of glass globes. In this case the fittings must be air-tight, or nearly so, as any current of air would cause the loss of iodine, which is volatile at all ordinary atmospheric temperatures. The iodine is deposited in small crystals, together with some water. The water may be drained and pressed off, and the iodine obtained in large dry crystals by a second sublimation.

#### QUESTIONS FOR EXAMINATION.

What is distillation?

In a solution of volatile liquids, what relation does the boiling point bear to the boiling point of its constituents?

In a mixture of liquids which do not dissolve one another, what will be the boiling point?

What will be the nature of the distillate from a solution or mixture of fluids having different boiling points? And how will the first and last portions of distillate differ from one another?

In what respect does sublimation differ from distillation?

What circumstances favour the massive or pulverulent condition of a sublimate?

#### RECAPITULATION.

Distillation essentially consists of a conversion of a liquid into its vapour, and the recondensation of the vapour into the liquid state.

The boiling point of a solution lies between the boiling points of its constituents: the boiling point of a mixture of fluids as about the boiling point of its most volatile ingredient. In distilling mixed fluids, the most volatile comes over most rapidly, but the less volatile come over more rapidly than they would do at the same temperature if the more volatile were absent. A solution of liquids of different boiling points may be approximately separated into its constituents by regu-

lating the heat of the boiler, or by using a series of condensers the temperatures of which are regulated.

Sublimation consists of the conversion of a solid into a vapour, and its reversion to the solid state. Volatile solids may be divided into those which volatilise more or less at ordinary temperatures and those which volatilise only at a high heat.

Sublimates are obtained massive by condensation on a surface not much cooler than the vapour. They are obtained pulverulent by condensation in a cold atmosphere.

## LECTURE X.

### FILTRATION AND PERCOLATION.

Filtering Media, Paper, Calico, &c.—Means of Expediting Filtration—Continuous Filtration—Pressure Filters—Filter Baths—Filtration through Charcoal—Percolation, Theoretical Considerations—Comminution Required—Forms of Percolators—Displacement of the Absorbed Liquors—Strong Tincture of Ginger—Modes of Packing the Material—Conical Percolators—Displacement of Spirit by Water—Pharmacopœial Process.

FILTRATION is one of the many simple operations of pharmacy, which at first sight appears to require scarcely any comment; nevertheless, there is scope for the exercise of intelligence and ingenuity in its many useful modifications.

The process itself may be defined as the passing of a liquid through a porous solid, and its object the separation of suspended or dissolved matter. The solid may be a colander, sieve, or cloth of open texture, in which case the separation of large particles only can be effected, and the process is, for distinction, called straining. It does not separate fine particles, or make a turbid solution bright. The filtering medium may be closer, such as a close calico, flannel, felt, paper, sand, pumice stone, asbestos, &c., when the object is to separate fine suspended matter, or charcoal, if the object is to separate matter held in solution.

Paper filters, with which you are all familiar, are made of coarse unsized paper for galenical purposes, or of fine unsized paper for fine precipitates. The coarse paper generally contains a mixture of wool, and is not pure enough for analytical use, but is tougher, and filters many pharmaceutical liquids both rapidly and well. From its thickness, there is more loss by absorption, but as it permits a rapid passage of the fluid, there

is little loss by evaporation. It is usually tough enough to bear the weight of a pint or two of liquid, while the thin paper would be burst in the throat of the funnel by such a volume. It is sometimes desirable to use a fine paper within a coarse one, thus combining the toughness of the strong paper with the fine filtration of the thin paper. A greater rapidity of filtration is thus obtained than if the fine paper only had been used. At other times a cone of perforated zinc may be put into the funnel, and the fine paper laid upon it, taking care, however, that such arrangement be not used for any liquid having a chemical action upon the zinc. Filtration will go on much more rapidly if the funnel is well selected as regards its shape, or the paper carefully folded to suit the funnel. If a circular paper be folded across its diameter, and then again at right angles, and opened out to form a cone, it represents the shape of a well-formed funnel. But if this be put into a common glass or earthen funnel, it will often be found that the funnel is either a more acute or a more obtuse cone, or that the sides are not quite straight, in which case the paper is apt to give way, or the flow of liquid to be impeded. I prefer to have the paper folded, so as to produce a cone very slightly more obtuse than the funnel, in order to throw as much of the pressure as possible upon the upper part of the funnel, the paper lying rather loosely upon the funnel at the lower parts. The difference, however, must be only slight, or the funnel will not sufficiently support the paper, and rupture will be risked. Various other modes of folding have been suggested, but none offering anything like general advantages.

The calico strainer and straining stand is the next most common and most useful filter for our purposes. I have already described it in speaking of precipitates, and I have now only to add that a variety of materials may be used instead of calico, such as linen, flannel, felt, or paper laid upon calico, according to the nature of the fluid, or the precipitate. If paper is to be used, it may have a few plaits folded at the edges, so as to give it a concave form, approaching the shape the strainer will

assume when stretched with the weight of the liquid. It should then be put in its place and filled with water, to make sure that it adapts itself without bursting. Whatever may be the nature of the medium adopted for filtration, if the fluid contain sediment which deposits upon the filter, the sediment itself becomes part, and not unfrequently the most efficient part, of the filtering medium, but at the same time retarding the operation, it may be, to an inconvenient degree. Various devices have been adopted to expedite filtration when inconveniently slow. The principal causes of variation in the rate of filtration are as follows :—The filtration is quicker the more porous the medium ; the thinner the stratum of the medium the greater the pressure of the liquid upon it ; the greater the freedom of the liquid from sediment, which might choke the medium, and the greater the mobility of the liquid.

It is obvious that some of these circumstances do not admit of change at our pleasure, others only to a small extent. Thus the porousness or openness of texture of the filtering medium will depend upon the fineness of the sediment to be separated ; but we may at least avoid the use of a close texture when an open one is all that is required. Then, as regards the choking of the filter in any case where this is apt to occur, we may in some measure obviate it by allowing subsidence to take place, and decanting as much as possible of the fluid into the filter in a more or less clear condition, that there may not be a large bulk of fluid to filter after the medium has become choked with deposit. If slowness of filtration results from viscosity of the fluid, it may in most cases be accelerated by heat. This is very noticeable in the filtration of oils ; but if the object be to separate the frozen portion of the oil from that which has a lower freezing point, the application of heat would defeat the object in view. The principal contrivances which have been devised for expediting filtration depend upon the increased pressure which they maintain upon the fluid in contact with the filtering surface. The contrivances for maintaining a given temperature, though they may be used to expedite filtration,

have been produced with the object of preventing crystallisation, solidification, or other change in the liquid which might result from a fall in the temperature.

I will now draw your attention to some forms of pressure filters suited to pharmaceutical use, and then revert to filter baths. The first, the simplest, and most generally applicable of aids to rapid filtration is the automatic arrangement for continuous filtration. For small quantities, nothing more is necessary than to invert the bottle containing the liquid to be filtered, supporting it upon a retort stand, with its mouth in the liquid, at the level to which it is desired to keep the filter filled. The liquid in the filter acts as a valve, preventing

Fig. 22.

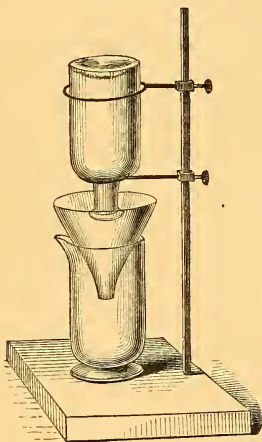
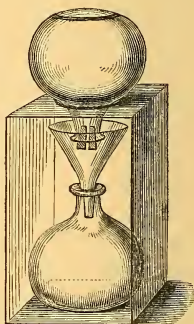


Fig. 23.



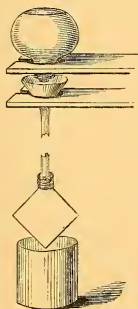
ingress of air, without which the flow of liquid from the bottle to the filter cannot take place. As the liquid in the filter sinks below the level of the bottle-mouth, a bubble of air enters and a corresponding portion of liquid flows into the funnel, so that the process is kept going on continuously, and as quickly as the pressure of a funnelful of liquid can make it, till the bottle is empty. To avoid splashing and waste of the contents of the



bottle, it may be fitted with a perforated cork to restrict the flow, which might be inconveniently rapid during the inversion of the bottle. For quantities of  $\frac{1}{2}$  gallon, or upwards, it is desirable to have a more substantial arrangement, as shown in the following diagram. A box with an open front has a hole made in the top, through which passes the neck of an inverted carboy containing the liquid to be filtered; inside the box a second carboy is placed to receive the filtrate, the filter consisting of the usual funnel and paper, supported by the lower carboy at such a level that the mouth of the upper carboy comes within the funnel a little below the level of the edge of the filter paper. Operating with vessels of this size, it is most convenient to have the upper one stopped with a perforated cork, the perforation itself being loosely plugged with a cork which can be removed when the apparatus is in working position.

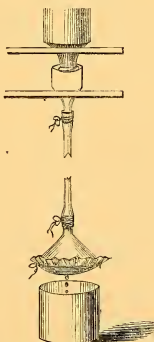
The next diagram represents a filter I adopted some years ago for filtering castor oil. It consists of a funnel, with a tube about 5 feet long. It is made of tin plate, with a ring soldered

Fig. 24.



B. S. P.'s Oil Filter.

Fig. 25.



B. S. P.'s Filter for Syrup, &amp;c.

round the bottom of the tube, to enable a filter-bag to be securely tied on. The bag is made of linen of a close texture,

and is placed over a jar to receive the filtrate, the funnel being supported by a projecting ledge from a high shelf, and kept full of oil by an arrangement similar to the last. The pressure of the column of 5 or 6 feet of oil causes the filtration to go on pretty rapidly, and if the cloth be of close texture the result is satisfactory.

The next diagram shows another arrangement upon a similar principle which I devised for filtering syrups, &c. It consists of two tin plate funnels, connected by an india-rubber tube. The upper having the same arrangement for keeping it full, the lower being of an obtuse form, and having the filtering

Fig. 26.

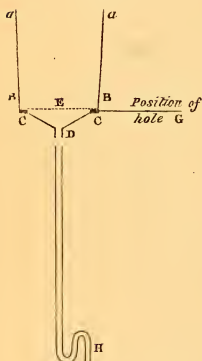
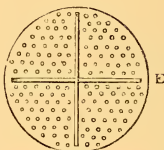


Fig. 27.



Perforated plate strengthened with cross bars below.

Fig. 28.



Ring with cross bars above.

Schacht's Filter. Conference 1865, Birmingham.

material stretched over its mouth, which hangs downwards over a jar which receives the filtrate. The filtering medium may consist of either cloth alone, or paper and cloth. In the latter case, of course, the cloth is put outside to support the paper. If a large quantity is to be operated upon, the funnels may be reversed, the narrow one being below, and having a filter bag attached, similar to that used for oil in the last arrangement.

One disadvantage of both these plans is, that at the conclusion

of the process a portion of the liquid has to run through without the assistance of the pressure ; and, unfortunately, that occurs at the time when the filter has become choked with deposit. Mr Schacht has obviated this objection by a system which he introduced at the Pharmaceutical conference in 1865. It consists of a tin funnel, with an india-rubber tube attached below. The funnel is made at two angles ; in the upper part the sides are nearly parallel, in the lower part the sides form about a right angle. Where these two parts join there is a ridge inside to support a perforated plate, and upon this plate is placed the filter paper or cloth, cut circular, and a little larger than the diameter of the funnel. The edges thus become turned up against the sides, and are kept in close contact with them by a tin ring, which is made so large that it just fits tightly into the bottom of the upper section, when the thickness of the paper or cloth is interposed between the ring and the sides of the funnel ; it thus lies upon and within the turned up edges of the paper, and prevents any of the fluid escaping filtration. The efficiency of this system depends upon atmospheric pressure. Under ordinary circumstances there is an atmospheric pressure upon the liquid in the filter, amounting to 15 lb on each square inch, but this is balanced by an equal pressure in the opposite direction, exerted through the opening in the neck of the funnel ; but when the long tube is attached, the under pressure is diminished to the extent of the weight of the column of liquid contained in the tube. Professor Attfield has stated that there are other forces in operation ; but we have not at present time to enter into a discussion of this question, and I may just state that the efficiency of this mode is said to be as great as that in which the pressure column is above the filter, with the advantage that it does not cease to act till the last portion of fluid has been forced through, but in some respects it is not quite so simple in management.

Filter baths are used for keeping filters hot while hot saturated solutions are being filtered, or to prevent the solidification of

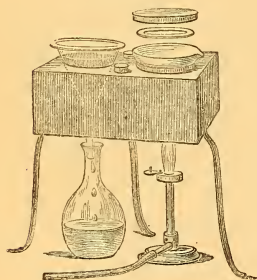
some body which has been liquefied by heat, such as lard, wax, ointments, &c.

The first figure represents a box of tin plate or copper, containing water kept boiling by a spirit lamp or gas flame below. There is a conical space to receive a funnel, and space in the top to receive a flask or beaker of the solution.

No. 2 represents a double funnel, the space between the two portions being filled with water kept hot by a spirit lamp applied to a projecting tube.

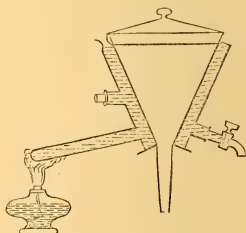
No. 3 is a funnel kept hot by a ring of gas jets placed below it, without the intervention of water. I have found it answer quite well for filtering ointments, &c., if carefully used, attention being paid to using as little gas as may suffice, and not bringing it in too close proximity to the funnel.

Fig. 29.



No. 1.

Fig. 30.



No. 2.

Filtration, with the view of removing substances held in solution, is generally effected by passing the liquid through a considerable stratum of animal charcoal, which has the power of absorbing or attaching to itself in some way many soluble substances. This property is possessed by other materials besides charcoal, though not to such a noticeable extent, and the action exerted by charcoal varies very much in degree, both according to the nature and purity of the charcoal, and according to the nature of the soluble matter to be removed.

The solution may be agitated in contact with the charcoal for some time, and then the whole filtered through paper in the usual way, or the charcoal

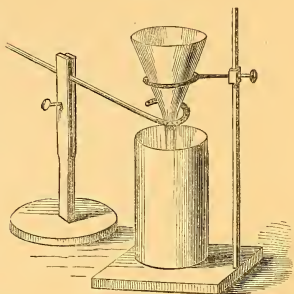
may be put into a percolator tube, and the liquid allowed to pass slowly through it. The animal charcoal of commerce is obtained by burning bones in close chambers, and contains a large quantity of phosphate of lime, from which it is necessary to purify it before it is fit for some purposes. The pure charcoal has more power of absorbing colouring

matters and alkaloids than that containing the phosphate of lime, and when it is necessary to redissolve an alkaloid which has been absorbed by the charcoal, of course the presence of phosphate of lime would interfere with the process. On the other hand, the charcoal becomes much softer and more dusty by the extraction of the bone earth, and as the dust is so fine as to pass through an ordinary strainer, it is customary to separate it from the granular portion by sifting, using the latter only for decolorising purposes. All these points have to be considered in determining whether the crude or the pure article should be used in any particular case. And it may be noticed in passing, that the purified animal charcoal of the Pharmacopœia has only a portion, though it ought to be much the greater portion of the earthy matter removed.

Percolation is very similar to filtration in its physical relationships, but is the converse of filtration in its object.

Filtration being a passing of a liquid through a solid, with the view of the solid *separating* something from the liquid, percolation is the same operation, with the view of the solid *imparting* something to the liquid. The solid body being reduced to a powder, rough or fine, according to its nature, and

Fig. 31.



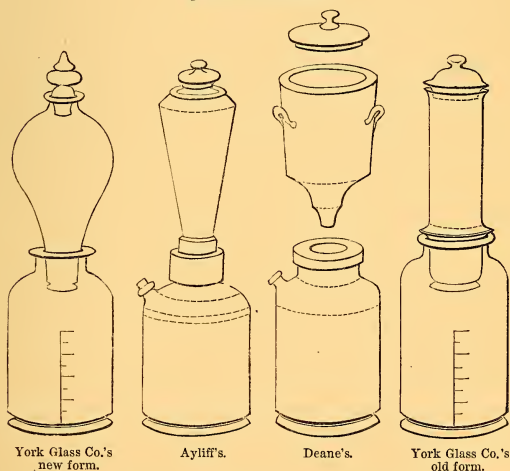
No. 3.

packed in a suitable vessel, the liquid is poured upon it, and, as it gradually descends, each particle comes in contact with a succession of different particles of the solid, and becomes more and more strongly impregnated with the soluble matter, till it escapes from the bottom. The solid particles also are brought in contact with a continual succession of fresh portions of the solvent, and part with some of their soluble matter to each, till exhaustion is completed, or till the supply of liquid ceases. Thus, in theory we might expect every portion of the liquid would become saturated before it passed through, and that exhaustion of the solid would be completely effected soon after the liquid ceased to pass through in a state of saturation. This theoretical result is most nearly obtained when the comminution of the solid is carried to its utmost limit, and the column is of considerable depth in proportion to its width. But, even under the most favourable circumstances, these results are far from attainable. The first portion of liquor is rarely saturated, and the exhaustion is never effected without the use of considerably more of the solvent than is capable of retaining the whole of the soluble matter in solution. Percolation is almost exclusively employed for vegetable substances the soluble portions of which are frequently colloid bodies, with only a small percentage of crystalloids, and if a saturated solution were obtained, it would usually have such a degree of viscosity as to stop the percolation; besides which, the solution of colloid bodies goes on with much less regularity and certainty than crystalloids. This viscosity impedes the percolation the more in proportion as the comminution is more perfect. It is, therefore, rarely practicable to use very fine powders, and the imperfectness and slowness of solution increase in proportion as the comminution is less perfect, so that the theoretical results are *never* obtained and *rarely* approached.

Fibrous materials which do not contain any large proportion of viscid matter may be reduced to the finest possible powder with advantage. Cinnamon, ginger, yellow cinchona, &c., cannot be too fine. Mucilaginous roots, such as rhubarb and

gentian, must be in a much rougher powder. Myrrh, opium, and similar substances are not readily exhausted by percolation, unless the most of the soluble matter has been extracted by a previous maceration, and decantation of the liquor from the insoluble; the exhaustion of the residue may then be finished by percolating a fresh portion of spirit. In all cases where the roughly bruised substances are to be operated upon, it is advisable to combine some form of maceration with the percolation. This may either be done, as in the pharmacopœial mode of making tinctures, by macerating the ingredients in part of the spirit for two days with agitation, and then transferring to the percolator, or by conducting both parts of the process in the percolator. For this latter method percolators are made with a provision for stopping the flow of liquid at pleasure; Mr Deane's

Fig. 32.—Percolators.



percolator being provided with a tap at the bottom of the tube. The York Glass Co.'s is made with the tube fitting air-tight into the reservoir by grinding, as a stopper is ground into a bottle, but with a flat portion or notch reaching a little more than half



along one side of each of the two ground surfaces, the notch on the receiver extending from the top of the ground surface a little more than half way down, and the notch on the tube extending from the bottom a little more than half way up. If these two notches are brought in juxtaposition there is an opening for the air to escape from the receiver, but in any other position the fitting is air-tight. The top of the tube is stopped, and notched in the same way. The percolation is stopped by making either of these junctions air tight, as the liquor will not run into the receiver unless there is egress for the air, nor will it run out of the tube unless there is ingress of air to the upper portion. We may call these adjustable joint valves. Supposing the lower valve to be shut, and spirit be poured upon the contents of the tube, it will only work its way down by capillary attraction, and by virtue of the compression the air undergoes from the weight of the spirit. If the lower valve be then opened the spirit will flow through, but may at any time be arrested to allow of maceration going on in the tube. Supposing it takes a half-pint of spirit to moisten the contents of the tube, and a half-pint more to fill the interstices between the particles, the interstitial half-pint will soon become strongly impregnated with the soluble matter, even though it does not penetrate into the cellular spaces of the vegetable to be exhausted—its impregnation depending upon the surfaces of particles exposing soluble matter, and upon the diffusion which takes place through the tissues of the vegetable where they are not ruptured. A third half-pint being poured into the tube, and the valve opened, the interstitial half-pint will run through, its place being taken by the new portion of spirit. But this action is not perfect; a portion of the original interstitial spirit may be retained in the mass; a portion of the intercellular spirit may have come through, together with a portion of the freshly added spirit. It is not to be expected that the new addition can precisely take the place of that which is draining out, without any mixture of the two taking place. We have evidence that considerable admixture

takes place, even under the most advantageous circumstances, as seen in the following experiments : A quart of fine sea sand requires a half-pint of fluid to fill its interstices. The stand being put into the percolator, and a half-pint of liq. potassæ permanganatis being poured upon it is entirely held by capillary attraction between the particles of sand. Here we have the interstitial half-pint without any intercellular liquor, or any soluble matter not already dissolved, to interfere with the observation, and have, consequently, the most favourable circumstances for obtaining a clear replacement of the liquor by a like bulk of pure water being added. But the addition of half-a-pint of water does not carry through the whole of the coloured liquor. A second half-pint of pure water being added to remove the first, drives out a highly coloured solution. A third measure of pure water being added, a pale solution percolates, but even now not absolutely colourless. This being the case with a material each particle of which is a smooth quartz pebble, how much more is it likely to be the case where each particle of the material is a porous mass like a sponge, or a fragment of wood ; and if the interstitial liquid is so imperfectly displaced, how much more difficulty must there be in displacing the liquid which has been absorbed into the cells and tissues of the material. This must necessarily take much time, and must depend greatly upon liquid diffusion, and this in the greater degree as the material is in larger particles. Consequently, in percolating roughly comminuted materials, it is desirable to add the spirit in separate portions, allowing each some time to macerate before it is displaced by its successor. There is probably no tincture for the preparation of which percolation is so clearly suited as the strong tincture or essence of ginger of the British Pharmacopœia. It may be taken as an illustration of what the process is capable of performing under favourable circumstances.

The official instructions are to pack the powdered ginger tightly in the dry state, and then to pour upon it a portion of the spirit equal to half the ultimate product. After this has

been in contact with the ginger for two hours, more spirit has to be added, and the percolation slowly continued till two fluid ounces of tincture have been obtained for each ounce of powder used. If the ginger be finely powdered, closely packed, and the percolator tube narrow in proportion to the quantity operated upon, so that the spirit has to percolate a depth of 10 or 12 inches, its downward progress becomes at last very slow, the tincture may be viscid from the quantity of resinous matter dissolved, and too thick to progress satisfactorily. This must be avoided by selecting the size and shape of the percolator so as to avoid such an excessive depth in the column. Some operators prefer to mix the powder for a tincture with as much spirit as reduces it to a creamy consistence, and in this condition pour it into the percolator, allowing it to pack itself, which it usually does sufficiently closely and in a more uniform condition than can be readily done by dry packing. It also avoids the excessive viscosity which I have just noticed as sometimes giving trouble, but it is better suited to cases where the quantity of spirit is greater in proportion to the dry materials, as in the formula under consideration more than one-half the spirit would be necessary to reduce the powder to the required condition, leaving an insufficient quantity for subsequent percolation to insure satisfactory exhaustion. A third method of packing is to rub the powder with just so much of the solvent as suffices to reduce it to the condition of a moist powder. It is said that this expedient prevents the chance of unequal percolation, which is sometimes observed in dry packing, the fluid passing down one side more freely than the other, and finding a channel of least resistance, resulting from some slight inequality in the packing. The testimony in favour of each method is so nearly equal that I am inclined to think all may be successfully worked by careful manipulation. My own experience has only led to conviction upon one point, namely, that the powder, if moistened beforehand, must not be worked into a paste; the quantity of fluid must either be so small as to result in simply moistening the powder, or so large

as to reduce it to a creamy state—the intermediate pasty condition almost certainly resulting in complete stoppage of the process.

I have also repeatedly observed that the material packed in the creamy condition contracts more uniformly towards the end of the process, and is less apt to crack in doing so, and thus leave open spaces. The form of the percolator is a matter of some moment, especially when the ingredients are packed dry, many substances swell considerably when moistened, and if they have not room allowed for expansion, become so tight that the liquid will not percolate at all. Others contract considerably when exhaustion is nearly completed, so as not to fill the space they were at first packed in. They then leave fissures through which the spirit will pass without permeating the mass. Both these contingencies are provided against by using a conical vessel. The contents of a funnel, if they expand, will rise in the middle without becoming choked, and when they contract, will sink into the funnel without leaving spaces. The column of material in a funnel is rather short in proportion to its bulk, but this is in part compensated for by the contents of the wide portion being always acted upon by the fresh spirit which has its solvent action unimpaired. Vessels prepared especially for percolation are usually of a more acutely conical form, such as those of the York Glass Co., or Mr Deane's, as made by Doulton and Watts. The form is of less consequence when maceration and displacement are combined, as directed in the *Pharmacopœia* for tinctures generally.

Attempts are sometimes made to economise spirit, by using water to displace the last portion, which would otherwise be retained by the exhausted material. What I have said regarding the impossibility of getting a perfect displacement of the first portion, by the second portion of spirit, will apply with greater force to the attempt to displace a pint of absorbed spirit by the addition of a pint of water. The disposition which water and spirit have to diffuse into one another, the difference in their gravity, which gives a tendency to the spirit to rise in

the water where they come in contact, the greater affinity which vegetable tissues have for water than for spirit, giving them a tendency to swell and choke the percolator, are all objections to the use of water for displacing the spirit; but these objections do not apply with equal force in all cases. I have succeeded with approximately good results in the case of powdered ginger, but when operating upon powdered cinchona, there was more appearance of mixture taking place,—a mixture which might readily escape notice, as it was not sufficient to make the tincture turbid; but at the same time, it must be admitted that it reduced the alcoholic strength of the tincture, and could not be considered legitimate in an official preparation. The directions of the Pharmacopœia now enjoining pressure after percolation, conveniently economises the great part of the spirit. If a more rigid economy is still desirable, distillation may be had recourse to.

I have already made several allusions to maceration, and need now only add that frequent agitation is necessary to make the process thoroughly efficient. The marc of a tincture, prepared by maceration and expression, will, of course, retain a certain small portion of tincture which no amount of pressure can expel, and in this case the loss must be a loss of tincture at least as strong as the bulk, and the only essential difference between the economy of the two processes is, that in many cases the loss, in case of percolation, is only a loss of spirit, or of very weak tincture, while the loss by the process of maceration is that of a tincture of full strength. The combined process now in use is superior to either separate system, in convenience, economy, simplicity, general applicability, and the quality of the results.

An arrangement of apparatus for the new process is described by Professor Redwood, in the "Pharmaceutical Journal" of May 1864, by which the necessity for agitation is avoided, the ingredients being suspended in a bag, from which they do not require to be removed till the processes of maceration, percolation, and expression are all complete.

### QUESTIONS FOR EXAMINATION.

Describe the nature and objects of filtration.

Describe the different arrangements for expediting filtration.

How does filtration through animal charcoal differ from filtration through most other media?

What precaution is necessary when an absorbed alkaloid is to be extracted from the charcoal by the subsequent use of an acid?

What is the essential difference between pharmaceutical percolation and filtration?

What materials are most suitably exhausted by percolation?

What kind of materials require fine comminution, and what kind require coarse?

How would you operate to make tincture of opium or myrrh by percolation?

Can the spirit absorbed by the marc of a tincture be satisfactorily forced through by percolating an equal bulk of water?

### RECAPITULATION.

Filtration consists of passing a liquid through a porous medium, with the object of separating suspended or dissolved matter.

Filtration may be expedited by arrangements for keeping the filter constantly full, for increasing the pressure of the liquid upon the filtering medium, and for maintaining an increased temperature.

Animal charcoal has the power of abstracting from solution many materials when held by feeble solvents, and of yielding them up to solution again when subjected to more powerful solvents. The presence of phosphate of lime in the British Pharmacopœia purified animal charcoal must be guarded against in some of these applications.

Percolation is the passing of a liquid through a porous and comminuted solid, with the object of extracting soluble matter. It is most suited to substances which are not of a mucilaginous character, and do not form viscid solutions with the required solvent. Woody materials may be finely powdered for percolation; substances yielding a viscid solution should be more roughly comminuted, and should be macerated in a portion of the solvent before being submitted to percolation. For roughly comminuted materials alternate maceration and percolation is most advantageous. The successive portions of solvent added do not, under any circumstances, precisely displace the portions which preceded them.



## LECTURE XI.

### OFFICIAL PHARMACY.

Pharmacopœial Preparations—Making or Buying—The Pharmacopœia—Mode of Studying—Official Powders in order—Official Pills—Proportion of Active Matters—Dose—Excipients—Essential Oils used—Mode of Mixing—Modification of Excipients—Official Pills in order.

#### THE PHARMACOPŒIA.

AN important part of the pharmacist's duty is the preparing of official compounds from the formulæ of the Pharmacopœia. Every one must decide for himself which articles he should make, and which he should buy ready made; but I may lay it down as a commendable rule, to make occasionally as many preparations as possible, and from a comparison of the home-made with the purchased preparations, to determine which may be bought when you have not leisure to make them. In the public discussion of this question, attention has been principally directed to the query, "Does it pay?" and if we were guided by pecuniary considerations only, we might promptly conclude that the work would be most economically performed by considerable division of labour, each pharmacist making only a very limited number of articles, and each buying and selling with his neighbours; but in this way each would be learned in his own branch, and ill-informed in others. It is most desirable that you should have such a practical acquaintance with pharmaceuticals prepared by yourselves, with every attention to accuracy, as would insure your detecting defects should they occur in purchased goods. Chemicals which are of a sufficiently definite character to be criticised without this sort of "personal acquaintance" may usually have their quality

satisfactorily guaranteed by subjecting them to the examination indicated in the paragraphs headed Characters and Tests, which are appended to the name and definition in the Pharmacopœia.

In the critical examination of any work, the method of procedure will be much influenced by the object of study, especially in a work like the Pharmacopœia, which may be studied in connection with the natural classification of the substances yielding medicines, which would be most suitable for the study of *materia medica*; or, according to their therapeutic actions, which would be most instructive to the medical student, or according to the nature of the process and the physical condition of the product, which is the classification most advantageous to the pharmacist, and according to which I now propose to treat of the most of its contents in as brief a manner as possible, and to notice in detail a few typical and a few exceptional cases in each division of my subject.

Though the number of drugs that are used in powder is almost countless, the number of official powders is not large, probably from the facility of extemporising almost any combination of powders that may be desired. Had our time been unlimited, we might profitably have devoted a little attention to powders, simple or compound, which do not appear in the official list, but we had probably better confine our consideration at present to the powders of the Pharmacopœia.

#### *Pulvis Amygdalæ Compositus*

Contains sweet almonds, sugar, and gum acaciæ. It is always desirable to use Jordan almonds, as the similarity in shape between sweet *Valentia* and bitter almonds is so great as to risk their occasional mixture, or even the substitution of the one for the other, a mishap which could not occur if Jordan almonds were habitually used. A few minutes' immersion in hot water, or a long maceration in cold, softens and expands the skin of the almond, so that it may be easily rubbed off between the finger and thumb. The shorter

the immersion the better, so long as the skin becomes soft and loose enough for removal. If the almonds absorb much moisture, they are more difficult to powder, and the powder, when obtained, is apt to become mouldy with keeping; consequently, we are instructed to dry the almonds with a cloth, and if they have been long in the water, it is also better to allow them to dry by exposure to the air. The instruction to rub them lightly to a smooth consistence can scarcely be followed literally if the operation be performed in a mortar. They require beating and heavy rubbing to reduce them to a smooth, pulpy condition, which is converted into a granular powder by a light rubbing afterwards. The powdery condition of the compound is further improved by the addition of the sugar and gum; these absorb the oil, which is always more or less set free before the almonds can be rubbed or beaten smooth. It is directed to be kept in a lightly covered jar; but, if well prepared, it will keep well in any kind of jar, but should not be kept in paper, as it would in that case lose part of its oil, and the remainder would become rancid.

*Pulv. Antimonialis*

Is now a definite compound of one part of oxide of antimony with two parts of phosphate of lime, instead of the rather uncertain product of a similar nature which was formerly obtained by calcining sulphuret of antimony and hartshorn shavings.

*Pulv. Catechu Comp.*

In this powder we have 4 oz. of catechu in 10, but as the kino and rhatany are both powerful astringents, we may say that the proportion of active astringents is 8 parts in 10.

There is a direction regarding the preparation of many of the pharmacopœial powders, that they are to be passed through a fine sieve, and finally lightly rubbed in a mortar. The object of this proceeding is to insure the absence of knots of any of the simple powders in the compound. Suppose, for example, that they were all separately kept in stock, finely

powdered, and that the catechu had run together into knots, even a long continued rubbing might fail to make the mixture perfect. Unless recourse was had to sifting there would be little knots of catechu visible in the mixture. These knots would be broken by passing through the sieve, but as the other powders which did not happen to be knotted would pass through more freely, the portion first sifted would contain less than its legitimate proportion of catechu, and the last portion more. A light rubbing in the mortar, however, would suffice to make the sifted product quite uniform.

*Pulv. Cinnamomi Comp.,*

Also called pulv. aromaticus, now contains only cinnamon, cardamoms, and ginger in equal parts,—the long pepper which it formerly contained being omitted from the present formula.

*Pulvis Cretæ Aromaticus.*

This must not be confused with the last, which is an aromatic powder without chalk or sugar. It is also necessary to observe that it is much more aromatic than the old aromatic confection which it has displaced.

The nutmeg and cloves which it contains are not readily reduced to a fine powder by themselves, unless with the loss of a portion of their essential oils, but if rubbed with a portion of the sugar, the grinding is facilitated, and the sugar absorbs the oil. The colour also depends very much upon the mode of operating. If the instructions of the Pharmacopœia have been literally obeyed, the product is a pale buff powder; but manufacturers prefer to see the colour of the saffron developed, which is accomplished by rubbing the powdered saffron with twice its weight of water, and then adding some of the other powders, in preference the chalk and sugar only, and when thoroughly mixed, drying, re-powdering, and lastly, adding the spices. In this way a full bright yellow is produced. The quality of the powder cannot be judged by its colour. This is not even a guide to the proportion of saffron it contains,

upon which its commercial value greatly depends, and the medicinal value, which depends upon the aromatics and the chalk, is better judged of by its odour and taste.

*Pulv. Cretæ Arom. cum Opio.*

This is the same as the last, with the addition of opium, of which it contains 1 part in 40.

*Pulv. Ipecac. Co., commonly called Dover's Powder,*

Contains 1 part of ipecacuanha, and 1 of opium in 10. The remainder being sulphate of potass, used to facilitate the division of the powerful drugs, and give bulk to the product.

This preparation being used mainly as a diaphoretic, it is sometimes prepared with nitrate of potass instead of sulphate, the former salt being supposed to increase its action upon the skin.

*Pulv. Jalapæ Co.*

This contains one part of jalap in three; but, unlike the last, the saline adds to its activity both as a purgative and diuretic.

*Pulv. Kino Comp.*

This is one of the various compounds in which opium is hidden under the "Co." Besides kino, it contains  $\frac{1}{40}$  of opium and  $\frac{1}{10}$  of cinnamon.

*Pulv. Opii Comp.*

This contains  $\frac{1}{10}$  of opium, combined with pepper, ginger, caraway, and a small quantity ( $\frac{1}{30}$ ) of tragacanth, which gives it a suitable consistence when mixed with syrup to form a confection. The confection contains so much syrup as to reduce the proportion of opium from  $\frac{1}{10}$  to about  $\frac{1}{40}$ .

*Pulv. Rhei Co.*

Is the rhubarb, magnesia, and ginger so well known under the name of Gregory's mixture, the proportion of the ingredients, however, being a little varied. The official proportions are

ginger, 1 ; rhubarb, 2 ; magnesia, 6 ; Dr Gregory's original form was ginger, 1 ; Rhubarb, 2 ; magnesia, 8. On the other hand, it is sometimes made with less of the magnesia, viz. in the proportion of 1, 2, and 4, respectively.

*Pulvis Scammonii Comp.*

Contains scammony, 4 ; jalap, 3 ; ginger, 1. It is consequently one-half scammony. The pure resin is not ordered in this case, though it might be used with equal advantage. The odour would in that case be different from the preparation with native scammony.

*Pulvis Tragacanthæ Compositus.*

This contains  $\frac{1}{6}$  of tragacanth. It is used principally for promoting the suspension of heavy powders, such as subnitrate of bismuth, when prescribed in mixtures. It mixes with water more readily than the simple tragacanth, the sugar dividing the gums, and rendering them less liable to run into knots. The gum arabic has the same object as the tragacanth, but is much less efficacious; and the starch being insoluble in cold water, may be regarded as a useless addition.

The comparative value of tragacanth and acacia for aiding suspension may be noted from the fact that tragacanth mucilage of the Pharmacopœia is as viscid as the mucilage of acacia, which contains 32 times as much gum—the former being 1 drachm in 10 ounces, the latter 4 ounces in 10.

PILLS.

The pill masses of the Pharmacopœia are in many cases evidently devised with the view of their being divided into 5-grain pills—the dose being usually indicated as 5 to 10 grains ; but the proportion of the active constituents are frequently not arranged in any simple numerical relation to 5 grains. Thus 5 grs. compound calomel pill contains 1 grain of calomel and 1 of sulphuret of antimony ; gamboge pill contains 1 grain gamboge in 5 of the *dry* materials. So of compound soap pill,

there is 1 grain of opium in 5 grains of the *dry* materials—the exact proportion of the active ingredient in a 5-grain pill being interfered with in these two cases by the syrup used in one case and the water in the other, to give the requisite pilular consistence.

There are various masses in which the active ingredient has some other numerical relation. Thus, in the aloetic masses, the proportion of aloes is 2 in 4; in aloes and assafoetida pill, the proportion of each ingredient is 1 in 4 of the mass; in blue pill we have 1 part of mercury in 3 of the mass; in lead and opium pills, the proportion is 1 of opium in 8, and 6 of acetate of lead in 8. In these instances the doses are, of aloes and assafoetida, 5 to 10 grains (though the proportion is 1 in 4); of blue pills, 3 to 8 grains (though the proportion is 1 in 3) and of lead and opium, 3 to 5 grains (though the proportions are 1 and 6 in 8); of aloetic pills, 5 to 10 grains (though the proportion is 2 to 4).

The most usual excipients are soap, confection of roses, and treacle. Soap is considered advantageous in promoting the solution or emulsification of aloes and other resinous drugs. Conserve of roses and treacle have both the property of retaining the mass in a moist condition; the former has the advantage over the latter in giving the mass that species of firm plasticity which enables the pills to retain the shape given to them.

Several of the masses contain essential oils, which are added with the view of correcting the griping tendency of the purgative, or of relieving flatulence. They also impart a characteristic odour. Thus we might recognise the pill of Barbadoes aloes by its odour of caraways, while the pill of socotrine aloes smells of nutmegs; rhubarb pill, of peppermint; and colocynth pill, of cloves. As a general rule, all the dry ingredients of a pill mass should be separately powdered and mixed together before the excipient is added. There is less certainty of uniformity if the excipient is added before the powders are mixed, and there would be great difficulty in making a smooth, homogeneous mass if a material like aloes or gamboge were



added in broken lumps. In some of the pharmacopœial formulæ this mode of procedure is inculcated, in others the instructions are less definite, and in several instances other methods are precisely indicated.

The selection of an excipient will be determined by several considerations. We must first consider whether it is calculated to promote or preserve the action of the important constituents of the pill, and not in any case to decompose or interfere with them. In the next place, we must consider whether the product is to be preserved in the massive condition, to be divided, or combined with other masses, as occasion may require, in which case the excipient should be such as to prevent it becoming hard or crumbly. Again, if the mass is to be at once divided into pills, our selection should be such as to insure that it would have a good plastic condition when first mixed, and that the pills should retain their shape and their solubility, though not necessarily their plasticity.

It is desirable, therefore, that the pharmacist should be allowed a certain small latitude as regards the excipients, provided that the proportion and virtues of the active constituents are not detracted from.

We will now run through the formulæ, noting here and there the object of special modes adopted, or the necessity for precautions not indicated.

*Pilula Aloes Barbadosensis.*

Barb. aloes in powder,	2 oz.
Hard soap in powder,	1 oz.
Are to be beaten up with—	
Oil carraways,	1 drm.
Confection of roses,	1 oz.

In this case you will observe the dry materials are separately powdered; but mixing before the addition of the excipient is not inculcated, and though advantageous, it is not specially essential in this instance.

*Pilula Aloes et Assafoetida.*

Socotrine aloes in powder, . . .	}	of each 1 ounce.
Assafoetida, . . .		
Hard soap in powder, . . .		
Confection of roses, . . .		

Beat all together until thoroughly mixed.

Here the aloes and soap are directed to be powdered, but we are left uncertain about the assafoetida. The fact that this gum, when it is dried and powdered, loses much of the essential oil upon which its efficacy depends, points to the conclusion that the massive assafoetida should be used; but the instruction is not so definite as it ought to be, considering the impracticability of getting commercial specimens of assafoetida free from small mechanical impurities, such as fragments of wood, &c., without either straining, or powdering and sifting. As I pointed out this want of explicitness to the Pharmacopœia committee before the final revision of their work, I conclude they probably felt the difficulty of deciding to order powdering in all cases when a fine sample of assafoetida would be injured by it, or to order it to be used massive when the commercial drug is not often fit to be so used. Thus we may feel ourselves at liberty to take advantage of the doubt, and according to circumstances proceed one way or the other, as we anticipate we can obtain the best results.

*Pilula Aloès et Ferri.*

Sulph. iron, . . . . .	1½ oz.
Barb. aloes in powder, . . . .	2 oz.
Compound powder of cinnamon, . . .	3 oz.
Confection of roses, . . . . .	4 oz.

Reduce the sulphate of iron to powder, rub it with the aloes and compound powder of cinnamon, and adding the confection, make the whole into a uniform mass.

Here we have the instructions full and satisfactory, leaving me nothing to add but a passing caution, that powders of crystalloid bodies, such as sulphate of iron, make the pill mass

very crumbly unless the salt be very finely powdered, and the mass made rather soft.

It may also be noted that soap cannot be used as an excipient in a mass containing sulphate of iron, &c., as decomposition would ensue.

The purgative action of this pill is greater than might be expected from the small quantity of aloes it contains, the activity of the aloes being increased by its combination with iron. The mass should be divided into pills as soon as formed, on account of its growing very crumbly by keeping. When required for dispensing, the dry materials are better kept mixed without the confection, and labelled thus :—

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<i>Pulv. pro</i>	
<i>PIL. ALOES ET FERRI.</i>	
13 grains of this, with	
8 grains confection of roses =	
—	
21 grains pil. al. et fer.	

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*Pilula Aloes et Myrrhæ.*

Socot. aloes,	.	.	.	.	2 oz.
Myrrh,	.	.	.	.	1 oz.
Saffron dried,	.	.	.	.	$\frac{1}{2}$ oz.
Confection of roses,	.	.	.	.	$2\frac{1}{2}$ oz.

Triturate the aloes, myrrh, and saffron together, and sift ; then add the confection of roses, and beat them all together into a uniform mass.

This formula gives us occasion to remark upon the use of the word “dried.” It being the universal custom to dry vegetable substances before they are powdered, it is unnecessary to say “saffron dried,” except it be to imply that the myrrh and aloes are not to be dried, because the word is not appended to them.

The aloes is not supposed to be in any way injured by being dried, and can scarcely be powdered in the moist condition in which it is imported. But the myrrh, if dried, loses some essential oil in which its medicinal properties (if it has

any) may be expected to reside ; and the process is probably contrived to avoid the drying of the myrrh.

*Pil. Aloes Socotrinæ*

Is very similar to pil. al. Barb., and requires no comment.

*Pil. Assafœtidæ Comp.*

Assafœtida,	.	.	.	.	}	
Galbanum,	.	.	.	.		
Myrrh,	.	.	.	.		2 oz.
Treacle,	.	.	.	.		1 oz.

Heat all together in a water bath, and stir the mass until it assumes a uniform consistence.

In this formula none of the ingredients are directed to be powdered, and from the mode of mixing we might conclude that it was most desirable to use them in their soft condition and trust to their melting ; but the myrrh remains so tough under the action of heat and treacle, that it is very difficult to make a smooth mass. It is desirable to powder all the gums as fine as possible, avoiding the drying of galbanum and assafœtida by rubbing them in frosty weather, and sifting to remove foreign bodies ; the myrrh may be powdered at ordinary temperatures, without drying, provided a very fine comminution be not required ; but it should be used at the same time, as it will agglomerate with keeping. The other gums also agglomerate, but as they are more fusible, that is of less moment, though it is most advantageous before adding the treacle, to have them all in as fine a state of division as is compatible with their good condition. The method of mixing ordered for aloes and myrrh may be adopted with advantage.

*Pil. Cambogiæ Comp.*

This contains gamboge and Barbadoes aloes, each 1 part in about 6 of the mass, the remainder being comp. cinnamon powder, soap, and syrup.

*Pil. Colocynthis Comp.\**

About 6 parts of this mass contain 1 part of colocynth pulp, and 2 parts each of Barbadoes aloes and scammony. It consequently contains less colocynth, less aloes, and more scammony than the compound extract of colocynth. It must be noted also that the commercial powdered colocynth is not pure colocynth pulp, but pulp and seeds all powdered together; and as the seeds are worthless, and constitute from  $\frac{2}{3}$ ds to  $\frac{3}{4}$ ths of the whole, it is important to see that they are separated before the colocynth is powdered. It may also be noted in passing, that Barbadoes aloes is used in the pill, socotrine aloes in the extract; also that scammony is used in the pill, and resin of scammony in the extract.

*Pil. Colocynth. et Hyoscyami*

Contains 2 parts of the colocynth pill, with 1 of extract of henbane. It is customary to prepare it with the dry ingredients of the coloc. pill, as the extract of henbane suffices to form a mass with them without any additional excipient.

*Pil. Conii Composita*

Contains ext. hemlock, with  $\frac{1}{5}$ th of its weight of ipecacuanha; a sufficient quantity of treacle is also directed to be added, but with an extract of the usual consistence, any addition is quite unnecessary.

*Pil. Ferri Carbonates*

Is a substitute for, and an improvement upon, the old compound iron pill. The quantity of conserve ( $\frac{1}{4}$ th of the weight of the saccharated carbonate of iron) appears small, but is quite sufficient; the sugar in the carbonate enabling it to form a plastic mass with much less excipient than would be required for the same weight of pure carbonate of iron.

\* In old prescriptions this is sometimes named "Pil. coloc. co. ex aloes Bbd. ppt."

*Pilula Ferri Iodidi.*

Fine iron wire,	.	.	.	40 gr.
Iodine,	.	.	.	80 gr.
Sugar,	.	.	.	70 gr.
Liquorice root powd.,	.	.	.	140 gr.
Distilled water,	.	.	.	50 minims.

Agitate the iron with the iodine and water in a strong-stoppered ounce phial until the froth becomes white. Pour the liquor upon the sugar in a mortar, triturate briskly, and gradually add the liquorice.

These instructions answer perfectly well if literally obeyed ; but if ten times the quantity be made at once, there would be some danger, the heat becoming so great as to volatilise both water and iodine. I have seen a strong 8-ounce bottle burst with a loud report from the violence of the action in making a similar preparation in about ten times this quantity. The action in that case, however, was still more violent from filings having been used instead of wire. The wire is preferable to filings on account of its greater purity. If it were required to produce a larger quantity at a time than here indicated, it would be desirable, after the action had freely commenced, to keep down the temperature by immersing the bottle in cold water till the chance of overheating had ceased, and then to promote the completion of the action, which takes place only slowly in the cold, by raising the water to the boiling point. The sugar retards the oxidation of the iodide of iron, and the liquorice gives the requisite firmness to the mass. As 80 parts of iodine produce 97·5 of iodide of iron,\* the proportion of iodide of iron to the moist mass is 1 in  $3\frac{1}{2}$ , or 1 in 3 of the dry material.

The solution of iodide of iron should be decanted from the undissolved wire while it is still warm ; for in the presence of so small a quantity of water crystals of hydrated iodide of iron are deposited on cooling.

\* I    I    Fe    Fe

127 : 80 : : 28 : 17·5. And  $80 + 17·5 = 97·5$  FeI.

*Pilula Hydrargyri.*

The mercury requires long continued rubbing with the confection before the globules cease to be visible; so much so, that the process is rarely undertaken by the retail pharmacist, though in other respects it is simple and easy enough.

This mass, like many others, will sometimes become inconveniently stiff and crumbly by keeping; its plastic condition may be restored by working it in a mortar, with a very small addition of water, or even, in many cases, without that addition.

There is said to be 1 grain of metallic mercury in a 3-grain blue pill; a small portion, however, is always oxidised. Some pharmacologists attribute the activity of blue pill entirely to the oxide of mercury it contains, and it has been proposed to improve the formula by substituting the black oxide for the metallic mercury. A change of this kind could not be prudently made without very careful investigation. It is not by any means proved that the finely divided metal is void of activity, or that the black oxide would be so satisfactory in its action, as long experience has proved the blue pill to be.

*Pil. Hydrarg. Subchlor. Co.*

Subchlor. mercury,	.	.	.	}	each
Sulphurated antimony,	.	.	.		1 ounce.
Guaiaacum resin,	.	.	.		2 ounces.
Castor oil,	.	.	.		1 fl. oz.

Triturate the subchloride of mercury with the antimony, then add the guaiacum resin and castor oil, and beat the whole into a uniform mass.

Castor oil is preferred as an excipient in this pill, because it gives adhesiveness and permanent softness.

The pills are apt to become dark with keeping; this is said to result from the calomel forming a black sulphuret of mercury by decomposition with the sulphuret of antimony, a reaction which I have not been able to confirm.

The proportion of calomel is intended to be 1 grain in a 5-grain pill; but as an ounce of castor oil is more than sufficient



to make 4 ounces of the powder into a mass, the pills are a trifle stronger. When the mass is made with the view of being divided into pills at once, castor oil is not the most eligible excipient, as the pills remain sticky and unpleasant. The following is an excipient I used for this pill long before castor oil became official, and though I have repeatedly tried the latter, I still give a preference to the mucilage.

*Glycerine Mucilage for Pills.*

Powdered gum tragacanth,	.	.	3 drs.
Glycerine,	.	.	9 fl. drs.
Water,	.	.	4 fl. drs.

Mix the gum and glycerine till smooth before adding the water.

Four ounces of the dry materials require exactly one ounce of this mucilage to form a convenient mass, which retains its plastic condition, its solubility, its retentiveness of shape, and a ready miscibility with other aqueous masses, if that were necessary.

*Pilula Ipecac. cum Scilla.*

Compound powder of ipecacuanha,	3 oz.
Squill in powder,	} of each
Ammoniacum in powder,	
Treacle, a sufficiency,	1 ounce.

Mix the powders, and beat into a mass with the treacle.

It is to be observed that this pill is made with compound powder of ipecacuanha, and consequently contains opium. The proportion of treacle required being about 2 oz., there is 14 per cent. of squill and 4·3 per cent. of ipecac. and opium.

In the table of opium preparations, the Pharmacopœia states that this pill contains opium in the proportion of "1 in 16½ nearly." This statement would be correct if no treacle were required; but with the quantity of treacle above indicated, the proportion of opium is about 1 in 23.

*Pilula Plumbi cum Opio*

Contains  $\frac{1}{8}$ th of its weight of opium, and  $\frac{8}{10}$ ths of acetate of lead.

*Pilula Quinæ*

Contains  $\frac{3}{4}$ ths of its weight of the sulphate.

*Pilula Rhei Composita.*

All the dry materials of this mass are directed to be used in powder, and it is the only article in the Pharmacopœia in the preparation of which *powdered* myrrh is used. As it is a pill for which there is a great demand, and is consequently made in large quantities, the injunction to mix the oil with the powders before adding the treacle is a desirable precaution. The quantity of treacle ordered is rather less than I usually find necessary; this, however, depends partly upon its thickness. I generally find five parts instead of four to be desirable, in which case the mass contains  $\frac{1}{3}$ th of its weight of rhubarb and  $\frac{1}{7}$ th of Aloes.

Glycerine is a good excipient when it is desired to keep this mass soft, but it must be noted that if glycerine alone be used the mass is rather hygroscopic, and the pills become too soft and sticky if not kept in a very dry place, to avoid which a mixture of two parts glycerine with one of water or spirit may be used.

*Pil. Saponis Co.*

Opium in powder, . . . . .	$\frac{1}{2}$ oz.
Hard soap in powder, . . . . .	2 oz.
Water, . . . . .	sufficient.

This contains one of opium in five of the dry material, or about one in six of the mass. The propriety of calling it compound soap pill when its activity depends entirely upon the opium, has repeatedly been questioned, but the old name is retained with the view of enabling the physician to prescribe opium without its presence being evident to the patient. Soap is not a good excipient for keeping this mass in a working condition, and for dispensing purposes it may be kept in better condition by substituting the glycerine mucilage for a part of the soap and water, thus,—

Opium in powder, . . . . .	$\frac{1}{2}$ oz.
Soap in powder, . . . . .	$1\frac{1}{2}$ oz.
Glycerine mucilage, . . . . .	1 oz. Mix.

*Pilula Scillæ Comp.*

Squill powder,	.	.	.	1 $\frac{1}{4}$ oz.
Ginger powder,	.	.	.	} each 1 oz.
Ammoniac powder,	.	.	.	
Hard soap,	.	.	.	
Treacle,	.	.	.	2 oz. or sufficient.

A 5-grain pill contains 1 grain of squill. You may observe that the Pharmacopœia complicates the statement of the proportion of squill by saying nearly "1  $\frac{1}{4}$  parts in 6."

EXAMINATION QUESTIONS.

What principles should guide you as to whether you should make or buy any particular preparation?

What is the object of the mixing ordered to be used after the sifting of a compound powder?

What is the normal colour of aromatic chalk powder? How does the commercial article differ in appearance, and in the mode of its production?

What general rule is desirable to be observed in the making of pill masses?

What rule should be observed in the selection of excipients for pill masses?

What difficulty is found in making crystalloids into pill masses, and how is that difficulty to be met?

What are the advantages and disadvantages of glycerine as an excipient?

RECAPITULATION.

It is advantageous to make occasionally all the galenical preparations that can be made well on the small scale for the sake of practical acquaintance with them.

Sifting a mixed powder, the components of which are not uniformly smooth, may cause a partial separation, and should be followed by a second mixing.

The normal colour of aromatic chalk powder is a dull buff,

but with the same composition it is obtained of a yellow colour by the use of moisture in the process.

The friable materials of a pill mass should be powdered and mixed before adding the excipient, that uniform mixture may be more readily obtained.

The excipient of a pill mass may be varied according to whether it be required to be immediately divided into pills, or preserved in the massive state ; in the former case a temporary plasticity and permanent solubility must be aimed at ; in the latter case it should permanently retain both qualities.

Crystalloids make crumbly masses with ordinary excipients ; they should be made soft and worked into pills immediately.

Glycerine is a good addition to many masses when it is desired to keep them plastic, but is too hygroscopic to be used without other excipients.

## LECTURE XII.

### OFFICIAL LIQUORS OR SOLUTIONS

Simple Processes—Solutions of Solids—Acetate of Ammonia, &c.—Processes a little more Complex—Solution of Chloride of Antimony, &c.—Solutions of Gases—Solution of Ammonia, &c.

#### THE LIQUORS OF THE BRITISH PHARMACOPŒIA.

THE preparations coming under this head are very various in their nature. They may be classified according to the mode of their production. First, those in which a solid is added to a liquid, and solution takes place without any further manipulation. In this case the chief point to which I have to draw your attention is the strength of the solution. Then solutions of solids, the processes for which are of a more complicated character; and lastly, solutions of gases.

#### *Liq. Ammonice Acetatis.*

One ounce of carbonate of ammonia makes a pint of the liquor; but as 58 parts of carbonate are equivalent to 76 of acetate, a pint of the liquor will contain  $1\frac{1}{3}$  ounce of acet. of ammonia. The chief point to be attended to is to get the solution neutral; to ascertain this point satisfactorily a few drops may be boiled in a teaspoon to drive off the carbonic acid before using the litmus paper, because free carbonic acid will redden litmus paper to almost the same degree as a small excess of acetic.

It is a common trade custom to prepare solution of acetate of ammonia of greater strength than the British Pharmacopœia to save bulk, and dilute the same with the requisite quantity of water at the time of use. However these solutions be pre-

pared, they should be compared with a standard to make sure of their not being deficient in strength. I will make this comparison with a sample prepared in my own establishment, from a formula calculated to give a solution eight times the usual strength. I take fl. ʒij. of this solution, and put it into a test-glass with fl. ʒxiv. of water, and a little burnt sugar to colour it brown. Into a similar glass I put two ounces of solution of acetate of ammonia prepared exactly according to the British Pharmacopœia formula. I now take a glass tube, and dip it to the bottom of the coloured solution, and then close the upper end of the tube with my finger while I carefully transfer it and its contents to the other glass; when the tube touches the bottom of the test-glass I take my finger off the top of the tube, and very steadily draw up the tube, leaving its contents standing like a column of brown smoke in the centre of the clear liquor. After a few seconds the brown column subsides to the bottom, showing that the concentrated liquor was stronger, though scarcely appreciably stronger, than it was calculated to be.

*Liq. Am. Citratis*

Contains 3 oz. citric acid in about 24 ounces of the liquor, or about  $2\frac{1}{2}$  ounces in a pint; but as 4 parts of citric acid yield 5 of citrate of ammonia, there will be a little more than 3 ounces of citrate of ammonia in a pint of liquor; that is, nearly 3 times the strength of the solution of the acetate; but the dose is given in both cases as 2 to 6 drachms.

*Liquor Atropiæ and Liquor Atropiæ Sulphatis*

Are simple solutions, containing respectively 4 grains of atropia and of sulphate of atropia in an ounce, a little spirit being used in the former preparation to promote the solution.

There are considerable discrepancies in the statements regarding the solubility of atropia in water, authorities varying in their results between a solubility of 1 in 200 and 1 in 500 at ordinary temperatures. On account of this sparing solubi-

lity, the Pharmacopœia directs the atropia to be dissolved in the spirit, which is a much more powerful solvent, and to add this solution gradually to the water with agitation after each addition to prevent precipitation, which may take place if the above precaution be not observed. Warm water may be used to avoid this separation; or if it has taken place, re-solution may be effected with heat.

The absence of spirit in the solution of the sulphate is advantageous when it is desired to apply atropia to the eye, as the spirit causes much smarting.

*Liquor Morphiæ Acet and Liq. Morphiæ Hydrochloratis.*

Each contains 4 grains of their respective salts in an ounce, solution being promoted by the addition of a small excess of acid and a little spirit. It must be remembered that the solution, according to the London Pharmacopœia, contains 8 grains to the ounce. It is commonly stated that the present strength corresponds with the strength of laudanum. But according to the Pharmacopœia standard for opium, 100 grains make 3 oz. tincture, and contain 6 to 8 grains of morphia, which equals 8 to 10 of hydrochlorate (303 parts of morphia being equal to 376 of hydrochlorate), so that  $1\frac{1}{2}$  ounce of tinct. may be said to equal 1 ounce of liquor as far as regards the *quantity of morphia it contains*; but the dose indicated in the Pharmacopœia would imply the reverse, namely, that 1 part of the tincture is equal to  $1\frac{1}{2}$  of the liquor, for they give 5 to 40 minims of laudanum, and 10 to 60 of liquor of morphia. If the activity of laudanum depend upon the morphia alone, and the dose of morphia be fixed as above, the dose of laudanum ought to be 15 to 40 minims; but it is impossible to fix in any very definite manner the absolute or relative doses of medicines, an approximation being all that we can look for; and preparations of uncertain strength require, for safety's sake, that the dose should be rather understated, besides which opium will sometimes contain more morphia than the British Pharmacopœia requires.



*Liq. Potassæ Permanganatis*

Contains 4 grains of the salt in an ounce of water.

It is better freshly prepared, as it deposits peroxide of manganese when long kept, especially if exposed to light, the solution, of course, losing a portion of its active properties. As this change takes place to a greater extent from contact with organic substances, it should not be filtered through paper, or kept in a bottle stopped with cork.

*Liquor Sodæ Arseniatis*

Contains 4 grains of the anhydrous salt in an ounce.

*Liquor Strychniæ.*

An ounce of this liquor contains 4 grains of the alkaloid, dissolved by the aid of a little hydrochloric acid and spirit.

Strychnia dissolves with still greater facility in acetic acid, which might consequently have been used with equal advantage in preparing its solution.

We will next take sundry solutions which are a little more complicated in their production.

*Liquor Antimonii Chloridi.*

Black antimony is boiled in hydrochloric acid, by which chloride of antimony and sulphide of hydrogen are produced. The native sulphide contains quartz, &c., which are insoluble, and iron (which gives the resulting liquor a yellow brown colour). The points to be attended to are, to avoid any vessel which is acted upon by the acid; to use a gentle heat at first, and a boiling heat when the evolution of gas becomes sluggish; and to operate in a good draught, as the sulphide of hydrogen is very offensive.

We may notice, in passing, that the affinities of antimony for chlorine and sulphur are nearly equal, as sulphide of hydrogen precipitates sulphide of antimony from an acid solution of chloride of antimony, and *strong* hydrochloric acid again

evolves sulphide of hydrogen, reconverting the antimony into chloride.

*Liquor Arsenicalis*

Contains 4 grains of arsenious acid in one ounce, dissolved by the aid of carbonate of potass, and boiling; the solution takes place only slowly, as the carbonate has not a powerful solvent action.

*Liq. Arsenici Hydrochloricus.*

This is the same strength as the liquor arsenicalis, and nearly three times the strength of liq. arsen. chlor. of the P. L. The old name has been abandoned, as it implied that a chloride of arsenic was formed, which is not at all probable, seeing that, if chloride of arsenic be mixed with a bulk of water, it is decomposed into arsenious and hydrochloric acids. The liquor, if evaporated, leaves a residue which is not freely soluble in water, and contains but little chlorine. The mode of operating is the same as for the last preparation, continued ebullition being necessary to effect complete solution.

*Liq. Ferri Perchloridi Fortior.*

Iron wire is dissolved in hydrochloric acid to form a protochloride of iron, which is next converted into perchloride by the addition of a further quantity of hydrochloric acid and a small portion of nitric acid.

The strength of the acids should be ascertained beforehand; and if they do not come up to the required standard, an increased quantity must be used to compensate for the want of strength. A deficiency of strength in the acids results in the solution containing a portion of protochloride, and the resulting tincture will deposit a basic salt. As the solution gives off a large volume of gas just as the operation is about complete, it is necessary to use a vessel capable of holding much more than the quantity of liquid operated on. The change that takes place may be represented thus—



and the suddenness of the evolution of  $\text{NO}_2$  is accounted for by this gas having the property of combining with the proto-salts of iron, so that the gas is not evolved till all the proto-salt has become saturated with  $\text{NO}_2$ ; then any further addition of nitric acid not only evolves the  $\text{NO}_2$  due to its own decomposition, but also that which was combined with the protochloride of iron. To avoid the inconvenience of this sudden effervescence, I have adopted the plan of adding protochloride in successive portions to the nitric acid, instead of in the reverse order, as usually pursued. This modification ensures the complete oxidation or rather perchloridation of each successive addition of the solution, consequently the  $\text{NO}_2$  is never combined, and cannot be suddenly and violently liberated.

The solution is to be evaporated down in a water bath, or otherwise below boiling point, as the salt is liable to decomposition by a brisk ebullition, chloride of iron and hydrochloric acid being volatilised.

The solution even after evaporation usually contains a notable quantity of  $\text{NO}_2$ . Chemically speaking, this is unquestionably an impurity, though it does not affect its medicinal action. If it is desired to avoid the presence of oxide of nitrogen, the protochloride may be formed as in the first stage of the pharmacopoeial process, and subsequently converted into perchloride by passing chlorine into it till it no longer produces a blue precipitate with red prussiate of potass. An excess of chlorine would be quite as objectionable as the presence of nitrous compounds, but it is more readily driven off by boiling. Solution of perchloride of iron containing no free acid cannot be boiled without undergoing partial decomposition, hydrochloric acid being given off, leaving peroxide of iron in solution along with the perchloride. By whichever method it be made, it is desirable to have the hydrochloric acid in slight excess. Test-paper cannot be used for indicating the acid, as neutral salts of the heavy metals generally redden litmus, and it is better to regulate the neutrality or

excess of acid present by arranging the proportion of materials and the strength of the hydrochloric acid in the first instance; this is the more desirable in the liquor of perchloride of iron, as it has the power of dissolving a considerable quantity of peroxide.

*Liq. Ferri Pernitrat.*

In this case one portion of nitric acid yields oxygen to the iron, while a second retains the oxide in solution. The quantity of nitric acid used is rather more than sufficient for both these purposes.

If there is any deficiency of acid, a basic salt is apt to be deposited on keeping.

*Liq. Ferri Persulphatis.*

In the preparation of this solution ordinary protosulphate of iron is peroxidised by the use of sulphuric and nitric acids, the method of operating and the nature of the changes being similar to those described in the second stage of the preparation of liquor of perchloride of iron by the pharmacopœial process.

*Liq. Hydrarg. Nitr. Acidus.*

This is a peroxidised nitrate of mercury with excess of acid. Had the nitric acid been largely diluted and kept cold, the product would have been suboxidised and much less soluble; the boiling at the conclusion expels any oxide of nitrogen which had been retained in the solution, and ensures the complete peroxidation, as is indicated by the subsequent testing with hydrochloric acid.

*Liq. Hydr. Perchlor.*

Contains half a grain perchloride in an ounce. An equal weight of chloride of ammonium is added, which facilitates the solution of the mercuric chloride; but it is not necessary for its solution, as an ounce of water is capable of dissolving

20 grains without any such addition; the object of the chloride of ammonium is to prevent the decomposition of the solution by keeping. A solution of perchloride of mercury exposed to sunshine evolves oxygen and deposits calomel; the presence of hydrochloric acid or chloride of ammonium prevents this decomposition.

*Liq. Iodi.*

20 grains of iodine are dissolved in 1 ounce of water with the aid of 30 grs. of iodide of potassium. This is about  $\frac{1}{3}$  the strength of the liniment, and about double the strength of the tincture.

The following table affords an instructive comparison of several officinal solutions of iodine.

Iodine being a very powerful drug, it is very important to discriminate carefully between preparations varying so much.

*Comparative Strengths of Solutions of Iodine.*

	Iodine in grains.	KI in grains.	Aq. oz.
Liq. Iodi. B.P. . . .	20	30	1
Iodin. co. P.E. . . .	8	32	1
Pot. ioidid. co. P.L. . .	$0\frac{1}{4}$	$0\frac{1}{2}$	1
Iodin. co. U.S. . . .	22	44	1
			Spirit.
Tinct. iodi. B.P. . . .	11	5	1
Linim. iodi. B.P. . . .	55	22	1

*Liq. Lithiæ Effervescens—Liq. Potassæ Effervescens—Liq. Sodæ Effervescens.*

Lithia water is directed to contain 5 grains in the half-pint bottle. Soda and potass each contain 15 grains. The pharmacopœial instructions are not intended to teach the art of soda water making, but to fix a definite strength which should always be attended to in waters for medicinal use. The water is directed to be filtered after the carbonate has been dissolved in it, to remove any lime which may have been precipitated. If the lime be not separated by filtration, it is

redissolved by the carbonic acid subsequently forced in, and may be detected by the use of oxalate of ammonia.

*Liq. Magnes. Carbonatis.*

Freshly precipitated carbonate of magnesia is more readily dissolved in an excess of carbonic acid than a carbonate which has been dried, but even the freshly precipitated salt varies according to its physical condition, the lighter precipitates being more soluble.

The speed of solution also depends much upon the pressure used. The Pharmacopœia does not indicate any particular pressure, but orders the pressure to be continued for twenty-four hours. A much shorter time is sufficient if a pressure of several atmospheres be used.

The solution is ordered to be further charged with carbonic acid after the excess of magnesia has been separated; this excess of acid in great measure covers the bitterness of the salt, and tends to preserve it in solution. The strength indicated is greater than I have found in commerce, and greater than can be kept without considerable deposit taking place.

*Liq. Plumbi Subacetatis.*

When normal acetate of lead is boiled in water with oxide of lead, a second equivalent of oxide enters into combination and solution. The second equivalent of oxide, however, is retained by so feeble a force, that it is liable to be precipitated as carbonate on contact with the air, or on dilution with water containing carbonic acid. The strong solution contains about 8 ounces of the salt in a pint. The dilute contains 2 drachms of the strong solution, or 48 grains of the salt, in a pint. Distilled water should be used in the dilution; but as distilled water contains carbonic acid, unless special precautions are taken for its exclusion, the Pharmacopœia directs the dilute solution to be filtered. It is difficult to obtain it bright, and impossible to keep it so in use; it is better therefore to pre-

pare it fresh when wanted, and if practicable to use distilled water which has been boiled to expel carbonic acid, and cooled out of contact with the air.

*Liq. Potassæ—Liq. Sodæ.*

These preparations are made by boiling the carbonates of the alkalis with hydrate of lime in an iron pan, and decanting the liquor without filtering. Tin, copper, lead, or enamelled pans are acted upon by the caustic alkalies, and should be avoided.

As these liquors also act upon filtering paper and cloth, subsidence is preferred to filtration for the separation of the lime, their great affinity for carbonic acid necessitates their being kept in well-closed vessels; and as lead glass is more attacked by them than hard green glass, the latter is prescribed for their preservation. The boiling promotes the complete decomposition of the alkaline carbonates, and the subsidence of the carbonate of lime, but also increases the solvent action of the alkali upon silica or alumina which may be present in the limestone, thus introducing them as impurities in the liquor. In making 1 gallon, 1 pound of carbonate of potass is used, or 28 oz. of carbonate of soda; yet the liquor of potass is nearly one half stronger, as the potass salt only contains about 16 per cent. of water, while the carbonate of soda, having 10 equivalents of water of crystallisation, is about two-thirds water.

*Liq. Zinci Chloridi.*

A concentrated solution of chloride of zinc is obtained by digesting zinc in a quantity of hydrochloric acid, insufficient to dissolve the whole of it. Towards the close of the process it is boiled, to effect the combination of the last portion of acid, and to reprecipitate copper, or any of the more electro-negative metals which may have been dissolved while the acid was in large excess. As iron is not separated in this way, the solution is filtered off and subsequently treated with chlorine,



by which any protochloride of iron in solution is converted into perchloride (an odour of free chlorine indicating that the action is complete); in this condition it is readily precipitated by double decomposition with carbonate of zinc, which is added in successive portions till it ceases to throw down any more peroxide of iron. After a second filtration, the liquor is evaporated in a porcelain or stoneware basin, by which means any free chlorine is driven off, and the bulk is reduced to the required standard.

Forty-four fluid ounces of hydrochloric acid, sp. gr. 1.16, equals 51 ounces by weight, and containing 31.8 per cent. of HCl, the weight of real acid is 16 oz., and as

HCl	HCl	Zin.	Zin.
36.5	: 16	:: 32.5	: 14.24

the quantity of zinc which should be dissolved. There is thus  $1\frac{3}{4}$  oz. of zinc in excess to allow for the impurities which it always contains. The quantity of chloride produced should be 30 oz. contained in the 40 fluid ounces of liquor, which results from the complete process.

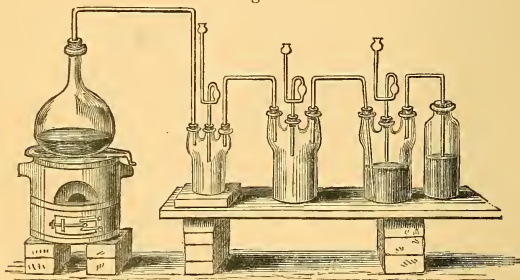
In the British Pharmacopœia formula you will observe the instruction to add carbonate of zinc in small quantities at a time to the solution of chloride, in the last stage of the process, "until a brown sediment appears." I should have said, until a brown sediment *no longer* appears to be thrown down by it.

In practice the official instruction will no doubt be generally sufficient, provided the solution of chloride of zinc be obtained by acting upon zinc with insufficient hydrochloric acid for its complete solution, because in this way no large quantity of iron is likely to be present, and the first addition of carbonate of zinc which gives any indication of iron at all probably precipitates all that is present; but if the same method be used for the separation of iron from zinc when the former is present in larger proportion, it is necessary to add successive portions of the carbonate of zinc till it no longer precipitates iron.

*Liq. Ammoniacæ—Liq. Ammoniacæ Fortior—Liq. Chlori.—  
Liq. Sodæ Chloratæ.*

These four solutions may be grouped together as depending for their formation upon the passage of gases through watery fluids at ordinary pressure and temperature. The ammoniacal liquors are by far the most important of the group; but as they are never made by the retail pharmacist, it is not necessary that we should devote much time to their consideration. The chief points of interest to us are to see that the solutions are of the requisite strength and purity. The gas is apt to carry over some mechanical impurities, which are deposited along with water and ammonia in the two first bottles (see fig. 33),

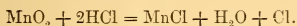
Fig. 33.



*Liq. Ammoniacæ Fort. B.P.*

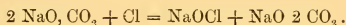
which take the place of the washing bottles, generally interposed between a gas generator and condenser. The extreme solubility of ammonia would risk the contents of the washing bottle being carried back into the generator, or with a rapid evolution of gas being carried forward into the condensing bottles. The two empty Woulf's bottles through which it passes serve also to cool the ammonia by which its solution is promoted. The third bottle is supplied with distilled water, and is sometimes immersed in a tub of cold water to remove the heat, which is liberated in considerable abundance, as the gaseous ammonia becomes liquid by solution.

Chlorine being much less soluble, is passed from the generating flask through a washing bottle, and then direct into the water, or solution of carbonate of soda required to make the respective liquors. The flask contains black oxide of manganese and hydrochloric acid, the decomposition being as follows—



The chlorine carries over with it a portion of hydrochloric acid, and some mechanically attached chloride and oxide of manganese, which are removed by the washing. It is pure enough for ordinary purposes

as it passes on to the absorbing jar. The combination that here takes place is not well understood, and the official name has been selected to avoid implying any theoretical constitution which has not yet been established. The following equation may be regarded as a probable statement of the changes that take place :—



It must not be inferred from the Latin name of this liquor that any chlorate of soda is formed in the process.

#### EXAMINATION QUESTIONS.

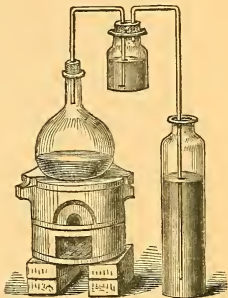
What rules are observed regarding the strength of some of the official solutions ?

What are some of the most notable exceptions ?

What objection is there to the present formula for solution of carbonate of magnesia ?

What precautions against the introduction of impurities are to be observed in the preparation and preservation of the solutions of the caustic alkalies ?

Fig. 34.



Liq. Sodæ Chloratæ, B.P.

In what respect might the instructions regarding the purification of chloride of zinc be modified with advantage?

#### RECAPITULATION.

Solutions of drugs which have not any great activity are of very various strengths. Solutions of alkaloids and sundry other very potent drugs are prepared in the proportion of 4 grains of the solid to 1 ounce of the liquor. Solution of perchloride of mercury is a conspicuous exception to this rule, its strength being  $\frac{1}{2}$  grain in 1 ounce. The solution of iodine is another notable exception, its strength being 20 grains in 1 ounce.

The solution of carbonate of magnesia is difficult to obtain of the standard strength, and more difficult to preserve of that strength.

The solutions of the caustic alkalies require caution to avoid their contamination with the material of the utensils in which they are prepared or preserved.

The official instructions for separating iron from chloride of zinc require a slight modification if the iron be present in considerable proportion; in the latter case say, "Add carbonate of zinc till it no longer precipitates oxide of iron."

## LECTURE XIII.

### OFFICIAL INFUSIONS AND DECOCTIONS.

Cases in which Infusion is preferred—Keeping Qualities of Infusions—Rules for Infusing—Temperature Varies—Time Varies—Comminution Varies—Decoctions, when preferred—General Rule and Exceptions—Decoctions of Aloes, Cinchona, Logwood, &c.—Sarsaparilla.

THE group of pharmaceutical products for our present study includes most of the pharmacopæial preparations in which the soluble part of a vegetable material is held in solution by water for use, while the insoluble and commonly inert matter is rejected.

The process of infusion is generally preferred in such cases where it is desired to extract volatile as well as fixed matters. Thus chamomile contains a volatile oil which would be partly volatilised and partly injured by boiling, but which imparts an aromatic flavour to the bitter extracted by infusing in hot water. The same may be said of orange peel, buchu, cloves, cascarilla, valerian, &c. The length of time infusion is to be continued varies with the solubility of the active matter and the condition of the material. The length of time that infusions will keep good depends much upon the presence of essential oils, and the absence of albuminous and mucilaginous compounds. Thus infusions of linseed, rhubarb, and calumbo are liable to become mouldy; but infusions of cinchona, cascarilla, and cloves, which are free from mucilaginous matter, and the latter two of which also contain essential oils, will keep a long time without any evident change.

The chief points to be attended to in making the official infusions are—

1. The exact temperature ordered.
2. The exact time, neither more nor less.
3. The infusion should be drained off without pressure.
4. The ingredients should be in a suitable state of comminution.
5. They should be stirred or suspended in the water, so as not to lie at the bottom all the time.
6. The ingredients of a compound infusion should each be weighed separately, not kept mixed in bulk.

Most of the infusions are made with boiling water, the exceptions being calumbo and quassia, which are made with cold, and chiretta and cusparia with water at 120° Fahr. (= 48·8° C.)

The temperature of 212° Fahr. (= 100° C.) coagulates vegetable albumen, if such be contained in the herb under operation; it also dissolves starch, which would not be taken up by cold water. The temperature of 120° Fahr. does not act upon the starch, and does not coagulate the albumen. We cannot always determine on theoretical grounds the most suitable temperature for any given infusion, the result being ultimately determined by experience.

The time required varies from a quarter of an hour to four hours; and, as I said with regard to temperature, I cannot point out theoretical grounds upon which the length of infusion could be determined; but I may note in passing, that it has recently been pointed out that the time of infusing may be reduced to one-half its present length without any loss of quality.

It is obvious that too short a maceration would imperfectly extract the active matters; but it may not be so readily perceived that too long a maceration may injure the product by dissolving an excess of mucilaginous matter, and this will be present in still larger proportion if the residue be pressed to get off the last portions of the infusion. The experiment may be tried on *inf. aur.* and *inf. rhei.*

The infusions to be macerated a quarter of an hour are four

in number, viz., anthemidis, aurantii, aurant compositum, and cusso. Those to be macerated for half an hour are seven, viz., caryophylli, catechu, chiratæ, ergotæ, maticæ, quassia, and rosæ. For one hour there are ten, viz., buchu, calumbæ, cascarillæ, digitalis, dulcamaræ, gentianæ compositum, krameriæ, rhei, senegæ, and sennæ. The infusions macerated two hours are six in number, viz., cinchona, cuspariæ, lupuli, serpentariæ, uva ursi, and valerianæ. Four hours' maceration is only ordered in one case, viz., inf. lini, and it is worthy of note that this is the only infusion which depends for its value solely upon the colloid matter it contains.

The degree of comminution is in most cases loosely defined, though the present is an improvement upon previous Pharmacopœias in that respect. Leaves and flowers do not generally require any comminution. Buchu is an exception to this rule, the varnish-like surface of the leaves rendering them almost impervious to moisture. The former instruction was to macerate the entire leaves for four hours; the leaves now being bruised, one hour's maceration yields an equally strong infusion; according to my own experiments, chopped leaves produced a better result than bruised, the infusion being brighter and less liable to mouldiness, probably because of the absence of particles capable of passing through the strainer in the cut leaves, and the presence of the same in the bruised.

The hard barks are to be reduced to a coarse powder, such as would pass through a sieve, 24 to 30 holes to the inch being suitable. Cinchona, cascarilla, and cusparia belong to this group. Catechu and cusso are also so ordered, though the latter may be supposed to be freely extracted without powdering, the object of comminution being to enable the patient to take the substance itself, as well as the infusion, for straining is directed to be omitted in this case.

Soft roots and herbs are usually directed to be cut small; this is quite sufficient in the case of orange-peel, calumbo root, chireta, matico, &c.

A degree of comminution between these two is indicated for



rhatany, senega, &c., by the word "bruised," which may be considered to express such a condition as would pass through a riddle having perforations  $\frac{1}{8}$ th to  $\frac{1}{4}$ th inch diameter.

Infusions which have been allowed to macerate without stirring, may be observed to be much darker at the bottom in contact with the vegetable matter, therefore occasional stirring should always be had recourse to during the maceration, unless the materials are supported in the upper part of the infusion pot in such a way as to allow of the water circulating through them.

I have already pointed out, in speaking of comminution, that several ingredients, mixed together in rough and unequal state of division, could not be depended upon for remaining uniformly mixed, and I need now only draw your attention again to the fact that, if the ingredients for compound infusion of gentian or orange-peel be kept ready for use, they should not be mixed in bulk, but each ingredient weighed separately into packets, each packet being enough for a single operation, say sufficient for a pint, or a quart, or a gallon of infusion.

The decoctions are aqueous preparations of such vegetables as are not readily spoiled by heat, and do not very readily yield their active principles to cold or warm water; there is perhaps, in some cases, no very strong reason for the selection of the one process in preference to the other.

The instructions for making decoctions are now much more satisfactory than they were in some of the former Pharmacopœias, the duration of the boiling being much reduced, and at the same time being made more uniform, the product being also made of uniform quantity by the addition of water to make up for what has been volatilised; so that the time and the quantity are now both definite, excepting in the single instance of decoction of pomegranate root, in which two pints of water are to be placed upon two ounces of the root, and boiled down to a pint, without regard being had to the longer or shorter time that it may require.

The general rule is to put on a pint of cold water, bring it

to the boil, and let it boil gently for ten minutes, afterwards percolating as much water through the residue as makes the product measure a pint. There are several exceptions to this rule; for instance, decoctions of poppy and sarsaparilla have a pint and half of water used in the first instance to produce a pint of the ultimate product.

In the case of sarsaparilla *boiling* water is poured upon the sarsaparilla, and an hour's maceration precedes the making of the decoction. The decoction of aloes, on the other hand, is macerated two hours after boiling before it is strained.

There are a few of the decoctions which required to be noticed a little more in detail.

*Decoct. Aloes Compositum.*

This is one of the most important decoctions in the Pharmacopœia.

It may be said to contain 4 grains of aqueous extract of aloes in each ounce, but as aloes always leaves a portion of insoluble matter each time it is dissolved and evaporated, we might naturally suppose that the sediment in the decoction contained the portion of the extract of aloes which had become changed and insoluble in water during the evaporation and drying of the extract. This, however, is not the case, the portion of aloes which had become changed and insoluble in water dissolves readily in water aided by carbonate of potass; the more important question is, whether it retains the activity which should belong to this weight of aloes. The discussion of this point will be taken up in a subsequent lecture on the pharmacy of aloes. Though the carbonate of potass promotes the solution of both aloes and myrrh, a considerable deposit generally takes place after straining and keeping for some time. This deposit is said to be inert; it should be separated by subsidence and decantation, as its slimy nature makes it rather difficult to filter. The saffron is only added after the boiling, but before the cooling. It is a common trade custom to prepare decoction of aloes four times the pharmacopœial

strength, it being stated that 1 part of the concentrated decoction mixed with 3 of water makes the decoction of the British Pharmacopœia. This, however, cannot possibly be the case, as the quantity of tincture of cardamoms would have to be 32 ounces, while the whole product would be only 30. It is not impossible that decoction of aloes might be improved, probably improved by its becoming an infusion in cold water or dilute spirit, instead of a decoction; but such questions are matter for the study and experiments of the authors of the Pharmacopœia, and whatever opinion we hold upon them, we are not justified in using as decoction of aloes any preparation but that made by the official formula.

*Decoct. Cinchonæ Flav.*

An ounce of this decoction is intended to contain the soluble matter of about 30 grains of bark. The decoction was formerly ordered to be strained while hot, it being supposed that the deposit which takes place on cooling contained alkaloids; but a closer examination having proved that it was only inert, resinous, and colouring matters,\* it is now directed to be strained cold, by which its appearance, at least, is improved.

Cinchona bark yields its alkaloids to water with considerable difficulty, especially if in coarse powder, the official process not extracting one-half of its active principles. There are no preparations from cinchona that can be considered satisfactory in a commercial point of view except the alkaloids, and if there is any ground for desiring any galenical representative of its entire activity, there is yet room for improvement.

*Decoctum Hæmatoxyli—Decoctum Granati—Decoctum Quercus.*

In preparing these and other astringent decoctions, care must be taken to guard against their becoming blackened by

\* Compare experiments on decoctum cinchonæ concentratum, &c., in a subsequent lecture.

contact with the pan if that be of iron. It is said, and I believe with some truth, that imperfectly tinned vessels are more likely to cause discoloration than clean iron, and that liquors may be boiled in an iron pan without injury, which would be blackened if allowed to cool in the same.

Decoction of logwood is very apt to get an inky tint from a very small contamination with iron ; its colour is also very sensitive to the action of various other chemicals ; not only will alum and iron salts deepen it, but even the carbonate of lime contained in many hard waters will make a visible change in its tint. This change will be seen the more readily for experiment, if you pour a few drops of the decoction into a glass of distilled water, and the same quantity into another glass containing water, which is hard from holding carbonate of lime in solution.

*Decoct. Sarsæ Comp.*

Jamaica sarsaparilla is the only kind acknowledged officially ; it is now ordered to be cut *transversely*, but not split as was formerly the custom. The bitter extraction residing only in the bark of the root, nothing was gained by splitting, which only facilitated the extraction of starch and mucilaginous matters which reside in the wood and pith.

QUESTIONS FOR EXAMINATION.

What kind of drugs are in preference extracted by infusion, and which by decoction ?

What qualities in drugs determine the comparative time they are to be infused ?

What constituents influence the keeping qualities of infusions for better or for worse ?

What materials require fine comminution for making infusions and decoctions ?

In what respect must the official decoction of aloes be essentially different from a concentrated decoction diluted with water ?

What precaution is necessary in making astringent decoctions ?

What kind of comminution should sarsaparilla root be subjected to in preparation for boiling, and what difference in the decoction results from the different modes of its division ?

#### RECAPITULATION.

Infusing is preferred to boiling for the extraction of drugs which contain valuable volatile matter, or are easily injured by heat.

The time required for infusion is determined by the compactness of the material, and the difficulty with which its active constituent is dissolved.

Essential oils improve the keeping qualities of infusions ; mucilaginous and albuminous matters detract from the same.

Porous substances only require coarse comminution ; hard barks should be finely divided.

Decoctions are preferred of such drugs as are difficult to exhaust, and are not easily injured by heat.

Concentrated decoction of aloes cannot be prepared to yield on dilution with water a fair representation of the official product.

The official process for decoction of cinchona only extracts about half the alkaloids present.

Astringent decoctions require care to avoid discoloration by the action of iron, &c.

## LECTURE XIV.

### EXTRACTS.

Varieties—General Process for Green Extracts—Extracts by Aqueous Infusion—By Decoction—Aqueous fluid Extracts—Liquors of Opium, Deane and Brady's, Battley's—Extracts Prepared with Spirit and Water—Alcoholic Extracts—Etherial Extracts.

THE extracts constitute a class of preparations very important in pharmacy, and of considerable diversity both in the nature of the process and of its product. The processes include infusion, decoction, percolation, expression, evaporation, distillation, &c., and the product is either a fluid, a semisolid, a hard mass, or a powder. They all agree, however, in being prepared in the fluid state first, thus naturally following the subject of my last lecture on infusions and decoctions.

For our present purpose they may be classified under three heads: those which are produced by expressing the juice of fresh plants, those which are obtained by the evaporation of infusions or decoctions, and those which are obtained by the use of alcohol or ether.

The general mode of procedure for the first of these classes is to collect the herb as quickly as possible early in the morning, to bruise it in a large mortar, or under a mill-stone, and then to press out the juice with a screw press such as is used for pressing the residues of tinctures. The juice thus obtained is heated without loss of time to 130° Fahr. (= 54·4° C.) by which the green colouring matter separates; the liquor is then strained, the colouring matter being reserved to add to the extract at a later stage; the heat of the clear liquor is raised to 200° Fahr. (= 93·3 C.), by which the albumen co-

agulates, and is separated by a second filtration and rejected, it being found that the albumen promotes the decomposition of the extract if not separated.

The clear liquor is then evaporated till of a syrupy consistence, a water bath being used to avoid the risk of injury by over heating. The temperature of a liquid freely evaporating in a water bath is always a few degrees below boiling point, generally as many as 20° or 30° Fahr. at the beginning of the process, but approaching more closely as the viscosity of the liquid increases. The extracts of colchicum and taraxacum are to be evaporated in a water bath at a temperature not exceeding 160° Fahr. (=71.1° C.), but in the other cases the use of the water bath without this restriction of temperature is considered to give sufficient protection against injuries by heat until the syrupy consistence has been attained, at which point the green colouring matter first separated is added to the liquor, and the evaporation continued at a heat which is now to be regulated both by the bath and the thermometer, and is not to exceed 140° Fahr. (=60° C.)

The green colouring matter is found to give the extract a better consistence than it would otherwise have, and keep it in a better condition. The ultimate consistence of the extract should be a rather soft pilular condition; but it is not found practicable to fix very definitely the consistence; some extracts are apt to become hard by keeping, others if evaporated to a firm consistence reabsorb moisture. This is the case with the extracts of colchicum and taraxacum, while extract of lettuce, aloes, logwood, or sarsaparilla have the opposite tendency.

The extracts prepared by this method are aconite, belladonna, hemlock, henbane, and lettuce from the green herb, and colchicum and taraxacum from the corn and root, the two latter being parts of the plant which do not contain chlorophylle; the first filtration is not required.

The groups of watery extracts may be divided according to the mode of procedure, thus the extracts of aloes are produced by hot infusion; opium, calumbo, and liquorice, by cold in-



fusion. By hot percolation we have, extract of poppy and pareira. By cold percolation—rhatany, quassia. By boiling—chamomile, gentian, logwood.

*Extractum Aloes Barb.—Extractum Aloes Socot.*

The aloes being dissolved in hot water, allowed to cool and settle, the soluble portion is separated and evaporated to dryness in a water bath. It must not be supposed, however, that the aqueous extract thus obtained is entirely soluble in water, for the extract undergoes some change during the evaporation by which a portion is rendered insoluble, and however often the solution, filtration, and evaporation are repeated, this always continues to be the case. I have myself repeated the process till there was only  $3\frac{1}{2}$  per cent. of the aloes left soluble, and this was almost destitute of purgative properties. The extracts of aloes are not to be kept of a pilular consistence, but reduced to dryness.

No definite relation can be stated between the strength of aloes and the extract. I am disposed to think that good aloes is not improved by the process, but that samples of varying quality are made more uniform by the improvement of those which contain much insoluble matter.

*Extractum Calumbæ.*

Cold infusion is preferred for this extract to avoid dissolving the starch which is abundant in the root, and would add to the bulk, but detract from the quality of the extract.

*Extractum Glycyrrhizæ*

Is also prepared by repeated macerations in cold water alternated with pressure. The use of the press in this and similar cases is very important, as it enables the exhaustion to be completed in much less time and with much less liquor, thus saving evaporation and improving the flavour.

*Extractum Opii.*

The opium is macerated in successive portions of hot water, the liquors filtered off, and evaporated to a pilular consistence. It yields about half the weight of the original opium, and as it contains nearly the whole of the active matters it is double the strength of the crude drug. It is also to be noticed that the extract is free from the earthy odour which is so characteristic of opium and its tincture.

Most drugs lose a portion of their medicinal activity during drying or the evaporation of their extracts ; opium, under these circumstances, loses narcotine, but it is said that the morphia is not diminished.

*Ext. Papaveris—Ext. Pareiræ.*

These two drugs are exhausted by infusing in hot water, and afterwards percolating more hot water. They should not be in a very coarse state, or the percolation is too rapid. It is to be noted that the poppy seeds are to be removed from the capsules, the seeds being oily, mucilaginous, and nearly, if not quite, inert.

*Extractum Kramerie—Extr. Quassie.*

These extracts are prepared by infusing the roughly powdered drugs in cold water, and afterwards percolating cold water till exhaustion is effected, the liquors being evaporated in a water bath. The extract of rhatany is to be reduced to dryness, the quassie to the usual pilular consistence.

*Extractum Anthemidis—Extr. Gentianæ.*

In the preparation of these two extracts we have recourse to actual boiling of the ingredients ; in the case of gentian the boiling being preceded by a couple of hours infusing. Chamomile contains an essential oil which is dissipated in the process, but its loss is compensated for by the addition of oil of chamomile at the end of the evaporation.

*Extract of Logwood*

Is evaporated to dryness in copper or enamelled vessels, the contact of iron being avoided on account of the blackness which it occasions.

The aqueous fluid extracts are—bael, cinchona, ergot, opium, pareira, sarsaparilla.

The processes for these preparations are very similar to those for the firm extracts, except that the liquors are evaporated down to a given measure, or to a given density, and spirit is added to prevent mouldiness or fermentation.

*Extractum Belæ Liq.*

1 lb bael fruit is macerated in successive portions of water, and the liquors evaporated till they measure 14 fluid ounces, then 2 fluid ounces of spirit are added, consequently a fluid ounce of the fluid extract is equal to an ounce of the fruit.

*Extractum Ergotæ Liq.*

Moist ether is percolated through the powdered ergot to remove its oil. (This is probably an unnecessary proceeding, as the oil is not soluble to any perceptible extent in water.) The ergot is then exhausted by digestion in a considerable bulk of water, which is subsequently filtered and evaporated, so that the product of 1 lb of ergot measures 9 fluid ounces, 8 ounces of rectified spirit are then added, which causes the coagulation of mucilaginous matters, which are to be separated by filtration. The product from 1 lb of ergot should be 1 lb of fluid extract.

*Extractum Opii Liquidum.*

This is prepared by dissolving 1 oz. of *extract* of opium in 16 oz. of water, and adding 4 oz. rectified spirit. Though the extract of opium was a perfect solution before it was evaporated, it does not now perfectly redissolve. Messrs Deane and Brady have founded a new process upon the fact, that each evapora-

tion separates some portion of matter by rendering it insoluble, while it is supposed that the morphia salts remain unaltered. They take  $1\frac{1}{2}$  ounce of opium and exhaust it with cold water, evaporate the liquor till a soft extract is obtained, redissolve this in half a pint of water, subside and filter, evaporate again to the consistence of treacle, add 5 ounces rectified spirit, by which the mucilaginous matters are precipitated; the clear liquor being decanted from this coagulum, is made to measure 20 fluid ounces by the addition of water. Narcotine, resin, the odorous principle, and gum are thus separated.

The well-known liquors of Battley are prepared upon a somewhat different plan. In the "Pharmaceutical Journal" for 1850, Mr Battley gives a general sketch of his process. He advocates the use of cold water for extracting nearly all vegetable substances.

He recommends the material to be macerated in twice its weight of cold distilled water; making a second infusion with as much more water as is pressed off the first time, and after repeating the infusions two or three times in this manner, evaporating at  $160^{\circ}$  Fahr. to the sp. gr. of 1.2, and adding rectified spirit till the sp. gr. is reduced to 1.1.

He makes three observations in favour of the precise mode of operating which he advocates.

1. Water used in this way, he says, is capable of taking up almost all the matters that are soluble in either spirit or ether, resin, &c., being soluble in the presence of gum while the starch is left undissolved. According to his experience, cold water extracts are more aromatic, more transparent, more soluble, and less liable to decomposition, than those prepared with heat.

2. The drug being coarsely powdered and macerated in not more than twice its weight of water, repeatedly pressed, especially if of a soft and spongy nature, the maceration should continue for four or six hours, after which time the density of the liquor does not materially increase. If more water is used at a time, an excess of gum is dissolved together

with other inert matters, thus weakening the extract, also injuring the product by increasing the required evaporation.

3. Evaporation to a dense fluid is preferred to the production of a firm extract, because the separation of more water injures its solubility, excess of heat has a like effect, and when the constituents of the extract are brought together in a very concentrated condition, new combinations take place, and changes go on during the whole time the extract may be kept. Evaporation to a definite specific gravity he considers gives a nearly uniform product, even if the quality of the drug operated on varies from time to time, and the subsequent addition of spirit preserves it from decomposition.

The sciences of medicine and of pharmacy are probably best promoted by the use of pure alkaloids, whenever their chemical and therapeutic properties are sufficiently well known to permit their commercial production and medicinal use.

*Extr. Cinchonæ Flav. Liquidum.*

The Pharmacopœia process for this preparation may be considered a modification of Battley's, as the powdered bark is macerated in a little more than double its weight of water, and then exhausted by the percolation of more cold water. The subsequent evaporation is carried ultimately till a specific gravity of 1·2 is obtained, as Battley directs, and then spirit added in such quantity as to reduce the gravity to 1·1.

According to my own experience, it is impossible to obtain even an approach to a satisfactory exhaustion of the bark, by the percolation of 12 pints of water through 1 pound of the cinchona, even when very finely powdered.

*Extr. Pareiræ Liquidum*

Is prepared, like the solid extract, by hot percolation, and is evaporated so that 1 lb of the fluid extract shall represent 1 lb of the root.

*Extr. Sarsæ Liquidum.*

In this instance the water is used at 160° Fahr. The root is cut transversely, but not split, that the starch may not be so much dissolved, two macerations are used in the place of percolation, the evaporation is continued till a specific gravity of 1.13 is attained, and spirit subsequently added. It is expected that 1 lb of sarsaparilla will yield 8 fluid ounces of extract, but the density being fixed is a protection against the root or the process being at fault, as in that case the quantity rather than the quality would suffer.

*Exp.*—Test infusions of split and unsplit root with iodine, the former gives an abundant precipitate of iodide of starch, the latter little or none.

*Extr. Jalapæ—Extr. Lupuli—Extr. Rhei.*

These three extracts are produced by the joint action of spirit and water upon the drugs which give them their name.

Jalap is extracted with spirit first, and then with water, the two liquors being separately evaporated to the consistence of soft extracts, then mixed, and the evaporation continued till a pilular consistence is attained. The activity of the extract depends almost exclusively upon the alcoholic portion—the mucilaginous matter extracted by the water being useful mainly as an excipient for retaining the jalap resins in an emulsable or diffusible condition.

Extract of hops is prepared by the same method; but extract of rhubarb is obtained by acting on the root with spirit and water mixed, the spirit being one-tenth the quantity of the water.

The quantity of spirit used in extracting the hops is very small compared to the bulk of the material operated upon, and a powerful pressure is necessary to press out of the hops a satisfactory proportion of the pint and half of spirit used.

The alcoholic extracts are—*Extr. cannabis indicæ*, *colocythidis compositum*, *nucis vomicæ*, *physostigmatis*, *stramonii*.

Hemp, nux, Calabar, and stramonium form a group. Hemp is simply macerated with spirit, nux is boiled with spirit, Calabar bean is macerated and percolated, and stramonium is percolated with spirit after having had its essential oil removed by percolation with ether. It is correctly classed with the alcoholic extracts, and not with the etherial, as the matter extracted by the ether does not enter into the extract ultimately formed.

In the processes with spirit and ether, it is desirable to economise these valuable solvents by having recourse to distillation instead of evaporation in an open vessel, the still body being heated by a water bath to avoid scorching, and the distillation continued till a syrupy liquid is obtained, the completion of the evaporation being effected in an open pan heated by water, the evaporation of the last portion of spirit taking place more rapidly, and at a lower temperature, in an open pan than in the close still body.

*Extractum Colocynthidis Compos.*

This is the most popular extract in the Pharmacopœia, though it might more appropriately have been called colocynth pill, had that name not been already allotted to a different compound. The colocynth pulp is exhausted by being macerated in *proof* spirit for four days (all the other alcoholic extracts being made with *rectified* spirit), then this proof spirit tincture is submitted to distillation till the spirit has come over, leaving the extract of colocynth in combination with a water liquid containing but little spirit, and to this is added the extract of aloes, scammony, resin, and soap; and then the evaporation is continued till a pilular consistence is obtained, the powdered cardamoms not being added till towards the end of the process, that their aroma may not be lost by the continued action of heat. There are few of the extracts which are conveniently kept of a firm pilular consistence, the firm extracts being liable to become intractably hard. This is much the case with colocynth; even when it is required for making



compound colocynth pills, it is more convenient to have the extract reduced to dryness, powdered, and reworked with a small quantity of rectified spirit; it is thus both easier to manipulate, and produces pills which retain their shape better. It is to be regretted that the authorities do not sanction the extract in the dry state, as the powder is almost exclusively the article of commerce. The proportion of the active matters in this extract may be roughly stated as being the soluble part of 1 grain of colocynth pulp, with 2 grains of extract of aloes, and rather less than a grain of resin of scammony, in a  $4\frac{1}{2}$ -grain pill.

It should be noted that the pulp only, and not the rhind or seeds, is used, and that aqueous *extract* of aloes, and *resin* of scammony, are directed.

*Extractum Filicis Liquidum—Extract. Mezerei Etherium.*

The former of these is a purely etherial extract, and the latter an extract obtained by the action of ether upon an alcoholic extract. The mezereon bark being extracted by maceration with successive portions of spirit, the bulk of the spirit distilled off, and the residue evaporated to a soft extract, the ether is then added, which takes up only a portion of the matter soluble in alcohol, the etherial solution is then distilled, and at last evaporated to a soft extract.

In making extract of male fern the ether is made to percolate through the root in powder, and to economise ether, a still is contrived upon such a principle that the same portion of ether is percolated, distilled, and re-percolated, any number of times.

EXAMINATION QUESTIONS.

Describe in outline the process for extracts prepared from green herbs.

What precautions are used in the evaporation of the liquors for extracts?

What varieties of physical condition do the official extracts include?

What kind of change commonly results from evaporation, and what drug is said to be an exception to the rule?

What two extracts are prepared from fruits, from which the seeds are first to be separated?

How is sarsaparilla root divided in preparation for making extract, and why is the old custom discontinued?

Which two extracts are prepared by making alcoholic and aqueous extracts separately, and then combining them?

#### RECAPITULATION.

The extracts are vegetable preparations, liquid in the first stage of the process, and concentrated by evaporation to a denser liquid, a plastic mass, or a friable solid.

The aqueous liquors, more especially from green herbs, should be evaporated with as little loss of time as is compatible with a safe temperature; a water-bath is generally used, and the heat also regulated by the thermometer.

Most vegetables lose part of their activity by drying, and most vegetable liquors lose value during evaporation; opium is said to be an exception to that rule.

Extract of colocynth should be prepared from the pulp of the fruit only, the rind and seeds being first separated.

## LECTURE XV.

### SPIRITS, TINCTURES, WINES, VINEGARS, LINIMENTS.

Spirits of Wine—Official Spirits not Redistilled—Tinctures—Various Wines—Sherry and Orange—Medicated Vinegars—Liniments—Aconite and Belladonna—Sundry Liniments.

THE study of this group of liquids most appropriately commences with a few remarks upon spirit of wine. It is unnecessary to enter into its chemical history; but the customary modes of expressing its strength in commercial parlance, and the rules for converting one strength into others, must claim a little attention.

#### *Spiritus Rectificatus.*

Alcohol with 16 per cent. of water, known in commerce as spirit of wine, 56 O.P., its sp. gr. being, according to the British Pharmacopœia, '838. The principal points of interest to us at the present time are the relation between the strength and gravity, and the modes of converting spirit of one strength to other standards. I will not attempt to enter fully into the matter, but must notice that the Excise standards are a rectified spirit of 63 O.P. It is known for Excise purposes as standard alcohol, sp. gr. '825, and contains about 89 per cent. of absolute alcohol. The second standard is known as proof spirit; its specific gravity is '920, and contains 48·5 of alcohol. Any strong spirit is reduced to proof by adding water till 100 measures become 100 plus, the number over proof of the sample before dilution. This takes a number of measures of water a little greater than the number over proof, as a slight condensation takes place on dilution. Thus, if we take the "standard alcohol" of the

Excise, that is spirit 63 O.P., we shall have to add water to 100 measures of spirit till it becomes 163 measures at 60° Fahr. to reduce it to proof spirit. The reason for this arrangement is very evident, the duty being fixed at so much per gallon on proof spirit; the scale on the hydrometer is constructed so as to indicate any number over proof; it makes a very simple calculation to ascertain what duty is chargeable upon the strong spirit. Thus, with spirit of the strength I have supposed, the number of the gallons is to be multiplied by 1·63, to give the number of gallons of proof spirit to which it is equal, and consequently by multiplying this by the duty, the payment upon the whole is indicated; and if the strength were 54 O.P., the number of gallons would be multiplied by 1·54 with the same object. And for our pharmaceutical purposes, if we have to reduce the spirit of 54 O.P. to proof, we add water till one gallon measures 1·54, and so on, of any other strength.

The official rectified spirit has a specific gravity of ·838, or, by the Excise rule, is 56 O.P., and the instruction for its dilution to the weaker spirit of sp. gr. ·920, which is equal to Excise proof, is to add 3 pints of water to 5 of spirit, or, in other words, 60 parts of water to 100 of spirit by measure. This, allowing a little for the condensation which takes place, agrees very closely with the Excise rule.

We have not the same interest in spirit under proof, but may just notice, in passing, that the number under proof being taken as a decimal fraction, and multiplied by the number of gallons in hand, gives the subtrahend, and the difference between this and the number of gallons of under proof spirit indicates the number of gallons of proof spirit it contains, and for Excise purposes would show the amount of duty chargeable. Thus, if you have 8 gal., 20 U.P., the calculation would be— $0\cdot20 \times 8 = 1\cdot6$ , and  $8 - 1\cdot6 = 6\cdot4$ , which is the number of gallons of proof spirit contained in 8 gallons of 20 U.P.

The statement which is current, that 20 U.P. spirit requires the addition of 20 measures of standard alcohol to 100 measures

to make it of proof strength, would be nearly correct if we substitute absolute alcohol for standard alcohol, or would be correctly put by saying, to 100 measures of 20 U.P. spirit add as much standard alcohol as would make 20 measures of water into proof spirit.

Spirits or alcoholic solutions, such as do not involve the use of distillation, may be considered next. They are—

Spirit of ether,	fl. oz. 1	of ether to fl. oz. 2	of rect. sp.
cajeput,	fl. oz. 1	of oil to fl. oz. 49	do.
camphor,	oz. 1	to fl. oz. 9	do.
chloroform,	fl. oz. 1	to fl. oz. 49	do.
juniper,	fl. oz. 1	of oil to fl. oz. 49	do.
lavender,	fl. oz. 1	,, to fl. oz. 49	do.
peppermint,	fl. oz. 1	,, to fl. oz. 49	do.
nutmeg,	fl. oz. 1	,, to fl. oz. 49	do.
rosemary,	fl. oz. 1	,, to fl. oz. 49	do.
Essence of aniseed,	fl. oz. 1	,, to fl. oz. 4	do.
peppermint,	fl. oz. 1	,, to fl. oz. 4	do.

In all these preparations rectified spirit is used ; in all the aromatic spirits (except, of course, aromatic spirit of ammonia) there is simply 1 part of essential oil in 50.

Spirit of chloroform may be regarded as a substitute for the old so-called chloric ether, which is simply a solution of chloroform in spirit, in somewhat uncertain proportions, and combined with small quantities of the by-products. It is said that the presence of these by-products increases the solubility of the chloroform in water, on which account the product of the old formula is sometimes called *soluble* chloric ether, as a further distinction between it and the similar preparation obtained by mixing chloroform and spirit of wine.

It is more likely, however, that the greater solubility simply corresponds to the smaller percentage of chloroform which it contains.

The old chloric ether was prepared by distilling spirit with chloride of lime in an insufficient quantity to decompose all the spirit, part being converted into chloroform, and part being distilled unchanged. These compounds must not be con-

founded with chloride of ethyl, known as hydro-chloric ether, or with the substitution products obtained by passing chlorine into ether, and known under the names of dichlorinated and trichlorinated ethers.

## TINCTURES.

Having spoken at some length upon the phenomena of diffusion of liquids in one lecture, and upon percolation in another, we shall have at present comparatively little to study under this head, but the composition, the proportion of active matters, and a few exceptional cases.

*Tinct. Aconiti.*

2½ oz. of the root in coarse powder is macerated in 15 oz. of rectified spirit for 48 hours, with agitation; the whole is then placed in a percolator, and when the spirit has drained through, 5 oz. more spirit is percolated; the marc is pressed; the liquors obtained by percolation and expression being all mixed, as much more spirit is added as is required to make the whole measure a pint.

This may be considered a typical process, several others being exactly the same, and a larger number only varying in some point of detail, such as the use of proof spirit.

The tinctures prepared by exactly this process are—

Aconite with rectified spirit.	Krameria with proof spirit.
Buchu with proof spirit.	Lobelia, do.
Calumbo, do.	Lupuli, do.
Cascarilla, do.	Myrrhæ with rectified spirit.
Chiretta, do.	Sabinæ with proof spirit.
Cinnamon, do.	Scilla, do.
Colchici sem., do.	Senegæ, do.
Conii, do.	Sennæ, do.
Cubebæ with rectified spirit.	Serpentariæ, do.
Digitalis with proof spirit.	Stramonii, do.
Gallæ, do.	Sumbul, do.
Hyoscyami, do.	Valerianæ, do.
Jalapæ, do.	Zingiberis with rectified spirit.

In the following tinctures the same proportion—viz., 2½ oz.

of the active constituent is used to produce a pint of tincture, but some other mode of extraction is followed.

Assafoetida—Is macerated in 15 oz. of spirit, filtered, and as much spirit added to the filtrate as makes it measure a pint. Rect. sp. 7 days.

Cocci, Proof sp. 7 days.

Limonis, do. do. Sliced peel.

Lobel. æther. Sp. of ether. 7 days' maceration.

Tolu, Rect. sp. 6 hours, or till dissolved.

Valer. amm., Ar. sp. am. 7 days.

This makes 32 tinctures, in which there is  $2\frac{1}{2}$  oz. of the crude drug to a pint of solvent, or an ounce of the tincture equals a drachm of the drug from which it is named.

A second list of tinctures may be drawn out, in which the proportion is 1 ounce of the drug to a pint of spirit.

Arnica root, Rect. sp.

Belladonna leaves, Proof sp.

Saffron, do. The process being maceration and percolation as used in the first part of the first list.

Indian hemp (ext.), Rect sp. Dissolve.

Castor, do. 7 days' maceration.

There are sundry tinctures of powerful articles, which are made with smaller quantities of their active material, and of some of which it is desirable to keep the proportion in mind.

Tinct. opii, . . .	33 gr. in 1 oz.
opii. ammon., . .	5 gr. „
camphora co., . .	2 gr. opium in 1 oz.
quiniaë, . . .	8 gr. in 1 oz.
cantharidis, . . .	5 gr. „
capsici, . . .	15 gr. „
aloes, . . .	11 gr. „

### *Tinctura Opii.*

Powdered opium is directed to be used in the preparation of the tincture. A deviation from the official process is sometimes made by estimating the quantity of opium in its moist state, which is equal to  $1\frac{1}{2}$  ounce of the powder, softening this down with warm water, and then adding rectified spirit



and water to make up one pint of tincture of the strength of proof spirit. The medicinal value of the tincture thus prepared is no doubt the same as that obtained by the pharmacopœial process, but its colour is paler, and its odour rather stronger.

*Tinctura Quinæ*

Will deposit some of the quinine in cold weather, unless some excess of acid be present, about 1 minum of diluted sulphuric acid to each grain of sulphate of quinine being a useful addition.

*Tinct. Card. Co.*

The seeds are to be freed from the pericarps and bruised before being weighed;  $\frac{1}{4}$  ounce is used for making a pint of tincture, and about three drachms of the entire capsules yield  $\frac{1}{4}$  ounce of the seed. Though cardamoms give the name to this tincture, it contains an equal quantity of caraways and double as much cinnamon.

*Tinct. Chloroformi Co.*

Contains 1 part of chloroform in 10, and is consequently double the strength of the spirit of chloroform, besides being one half compound tincture of cardamoms.

*Tinct. Cinchon. Comp.*

Is made with pale bark, 2 ounces in a pint, and is a warm aromatic tincture, but not rich in quinine.

*Tinct. Cinchon. Flavæ*

Is made with double the quantity of bark, and being a species which yields quinine abundantly (not less than 2 per cent. according to the British Pharmacopœia standard), it is a good tonic tincture, but not aromatic. The quinine it contains should be about  $1\frac{1}{2}$  to 2 grains per ounce, consequently about  $\frac{1}{4}$  the strength of tincture of quinine.

*Tinct. Ferri Acetatis.*

Acetate of potass and persulphate of iron are both soluble in spirit, but on being mixed double decomposition takes place, and sulphate of potass, which is insoluble in spirit, is precipitated, while acetate of peroxide of iron is retained in solution. By keeping, it acquires an odour of acetic ether, a basic acetate of iron being deposited, the tincture is paler and weaker. The addition of a small excess of acetic acid would probably be an advantage, and the tincture should be kept in the dark. The process might also be modified with advantage, by rubbing the acetate of potass with the liq. fer. before adding spirit; the precipitate thus produced is denser.

*Tinct. Ferri Perchlor.*

Is made by mixing one measure of strong solution of perchloride of iron with three measures of rectified spirit. A fluid ounce of the tincture contains about 62 grains of anhydrous perchloride of iron. It acquires an ethereal odour by keeping, and if prepared with an exactly neutral chloride, is liable to deposit a basic salt; this is avoided by having a slight excess of acid and taking care that no proto-salt is present.

Mr Squire suggests that this tincture may be prepared from the crystalline hydrate of perchloride of iron ( $\text{Fe}_2\text{Cl}_3 \cdot 12 \text{ Aq.}$ ) an ounce of which is equal to a fluid ounce of the official strong liquor. If this suggestion be acted upon, care must be taken to use the right kind of chloride of iron, as the commercial salt is usually  $\text{Fe}_2\text{Cl}_3 \cdot 6 \text{ Aq.}$ , with a little more water mechanically attached.

*Tinct. Guaiaci Ammoniata*

Contains 4 ounces of gum guaiac, dissolved in as much aromatic spirit of ammonia as will make the whole measure a pint.

*Tinct. Iodi.*

Contains  $\frac{1}{2}$  ounce iodine in a pint of rect. spirit, with  $\frac{1}{4}$  ounce iodide of potassium.

*Tinct. Lavandulæ Composita*

Contains 45 minims of oil of lavender in a pint; and turning to "oil of lavender," we find that the Pharmacopœia only sanctions the use of that distilled in Britain, from the flowers of *Lavandula vera*. There are many other instances in which such restrictions are in force, and not indicated where the article is named. It is necessary, however, to remember them, as in this case, when oil of lavender is ordered, that the English oil distilled from flowers of *Lavandula vera* only can be legitimately used. Oil of rosemary, which is also contained in this tincture, is ordered to be distilled from the flowering tops, but not necessarily in England. It also contains cinnamon bark and nutmegs, and is coloured by red sandal wood.

*Tinct. Nucis Vomicae.*

This is prepared from powdered *Nux vomica* with rectified spirit, by maceration and percolation in the usual manner, the proportion being 1 part of nux in 10 of spirit.

*Tinct. Rhei*

Is now made with a much larger portion of rhubarb than was formerly ordered, viz., 2 ounces instead of  $1\frac{1}{4}$  ounce to the pint; it also contains cardamoms, coriander, and saffron, and does not now contain liquorice.

*Tinct. Zingiberis Fortior*

Is the only tincture made by a pure process of percolation; it is four times the strength of the usual tinctures, viz., 10 ounces to 1 pint, and is used principally for making syrup of ginger.

## MEDICINAL WINES.

Very much resemble the tinctures, but are less alcoholic, the

proportion of spirit in sherry being from 17 to 20 per cent. of rectified spirit; the Pharmacopœia defines that it should contain 17 to 18 per cent. of alcohol, which is equal to about 20 to 21 per cent. of rect. spirit, and is about as strong as sherry can usually be obtained. Orange wine is directed to contain about 12 per cent. of alcohol, besides which, they both contain small quantities of extractive matter, vegetable acids, and salts. Bitartrate of potass being a normal constituent of grape juice, occurs in all the grape wines, though more abundantly in the new wines; as the fermentation goes on, the increase in the percentage of alcohol causes the precipitation of the greater bulk of the bitartrate; the small portion that is retained is useful in aiding the solution of the iron wire used in making the vinum ferri of the British Pharmacopœia; a small quantity of tannin, which it also contains, is the cause of the blackness.

The medicinal wines are—

Vin. Aloes—about 18 grains in 1 ounce.

Antimon.—2 grains in 1 ounce.

Colchici (Corm)—87 grains in 1 ounce.

Ferri—of uncertain strength, 1 ounce of wire being put in a pint of wine, but very little being dissolved.

Ferri citr.—8 grains citrate of iron and ammonia in 1 ounce (*orange wine*).

Ipecac.—22 grains in 1 ounce.

Opii—22 grains of extract in 1 ounce. The extract being about double the strength of opium, the wine is about  $\frac{1}{3}$  stronger than the tincture, which contains about 33 grains of opium in 1 ounce.

Quinæ—1 grain in 1 ounce (*orange wine*), the solution of the quinine being promoted by the addition of  $1\frac{1}{2}$  grain of citric acid.

Rhei—33 grains in 1 ounce.

Free acid is considered advantageous for the preparation of ipecacuanha wine. Mr Johnson, at the Pharmaceutical Conference at Birmingham, showed that the deposit which takes place in long-kept ipecacuanha wine contains ipecacuanhate of emetina, which is precipitated from ipecacuanha wine of any alcoholic strength, if there is no free acid; but samples pre-

pared with inferior acid wines were comparatively free from change with keeping. The addition of three or four grains of tartaric acid per ounce is an advantage in preventing this deposit.

Tartaric acid itself is not exempt from decomposition when kept in solution in water, and it is not unlikely that acetic or hydrochloric acids would be more advantageous. No doubt the spirit in the wine would retard the decomposition of tartaric acid, but acetic acid is not only more stable in itself, but also has an antiseptic action upon most organic substances.

#### VINEGARS.

Acetic acid being one of the best solvents for alkaloids, vinegar, or the pure acid diluted, may be advantageously used for extracting various vegetables, though the number of vinegars at present official is limited to two, viz., acetum cantharidis, which is prepared with strong acetic acid; and acetum scillæ, which is prepared with an acid of the strength of vinegar, with the addition of spirit.

Ordinary malt vinegar is official, though the only preparation in which it is directed to be used is the emplastrum ceratum saponis, in which an acetate of lead is extemporised by boiling oxide of lead in vinegar. Malt vinegar of the Pharmacopœia is directed to contain  $4\frac{1}{2}$  to 5 per cent. of acetic acid, and is allowed to contain a small portion of sulphuric acid, which, however, must not exceed 1 part in 1000.

#### LINIMENTS,

Though of very various compositions, may be classed along with tinctures for our present purposes.

##### *Linimentum Aconiti—Lin. Belladonnæ.*

These liniments are both prepared from the coarsely powdered root, by moistening 20 ounces of it with spirit, allowing it to macerate for three days, then packing it in the tube of a percolator, and putting 1 oz. of camphor into the receiver, and slowly percolating spirit through the root upon the camphor till the product measures a pint, thus an ounce

of the liniment may be considered to represent an ounce of the root, though it is far from containing all the active matter which exists in that quantity of root.

To promote the best practicable exhaustion of the material, the root should be so finely divided as to pass through a sieve, twenty holes to the linear inch, this rough powder being moistened with just so much of the spirit as suffices to damp it, not making it positively wet; it is then to be packed with considerable force in the percolator. This necessarily makes the percolation slow, but insures its being effectual, and avoids the loss of much spirit. The root, as usually found in commerce under the name of rough powdered belladonna, is not nearly so fine as desirable.

*Linimentum Ammoniac—Lin. Calcis—Lin. Hydrargyri.*

These are emulsive liniments containing imperfectly saponified grease.

The lin. ammoniac is olive oil partially saponified with the weaker liquor of ammonia. It is a thick fluid while fresh, but is apt to solidify by keeping, more of the oil being converted into soap than the remainder can retain in the liquid form. An excess of oil retains it in the liquid state more effectually than an excess of water, but different samples of oil vary in this respect.

Liniment of mercury is a similar compound, containing mercury and camphor.

Liniment of lime is prepared with equal parts of lime water and olive oil, not linseed oil, as has been for many years used. A lime soap is formed which is not soluble, but miscible with the oil, of which there is much more present than can combine chemically with the lime, but the whole forms with the water a thick creamy emulsion.

*Lin. Camphoræ—Lin. Camphoræ Comp.*

It is necessary to note the difference in these two preparations, it being not an uncommon thing in some neighbourhoods

to overlook the simple liniment altogether, excepting under the name of camphorated oil.

Camphor liniment is 1 part of camphor dissolved in 4 parts of olive oil. Compound camphor liniment is—camphor,  $2\frac{1}{2}$  oz.; oil of lavender, 1 drachm; strong solution of ammonia, 5 oz.; and spirit of wine, 15 oz.

Chloroform liniment consists of equal parts of measure of chloroform and *simple* camphor liniment.

Croton oil liniment contains 1 drachm of croton oil in the ounce, the remainder being equal parts of rectified spirit and oil of cajeput.

Iodine liniment, as I before noticed, in speaking of the tinctures, contains five times as much iodine as the tincture and four times as much iodide of potassium and a little camphor.

Linim. potassii iodidi cum sapone is a gelatinous mass obtained by dissolving—

Hard soap,	.	.	.	.	1½ oz.
Hot water,	.	.	.	.	7 oz.
And adding a solution consisting of—					
Iodide of potassium,	.	.	.	.	1½ oz.
Glycerine,	.	.	.	.	1 fl. oz.
Water,	.	.	.	.	3 oz.

And subsequently flavouring it with oil of lemon 1 fl. drachm.

Its gelatinous condition is a very questionable advantage, but being customary, it is well that all pharmacists should produce it alike.

The soap which is best adapted for the liquid liniment being least suitable for the gelatinous, common curd soap answers well. I have used that made in tablets by Anderson and Cattley, with perfectly satisfactory results.

### *Liniment Saponis*

Contains  $2\frac{1}{2}$  ounces of hard soap in a pint, and should be a clear pale yellow fluid. A small portion of the soap remains undissolved, if the temperature does not rise above 70° Fahr. in the maceration of the liniment; but if a higher heat be



used more of the soap dissolves, and the liniment gelatinises on the temperature falling. Much depends also upon the quality of the soap, even the olive oil soap not being all equally soluble; and though the Pharmacopœia directs that medicinal hard soap shall be soluble in rectified spirit, the precaution of keeping the temperature of the soap liniment below 70° Fahr. (=21° C) during maceration, is an additional provision to insure its not gelatinising in cold weather. There is also—

Camphor,	.	.	.	.	1½ oz.
Oil rosemary,	.	.	.	.	3 drms.
Rect. spirit,	.	.	.	.	18 oz.
Water,	.	.	.	.	2 oz.

To complete the formula.

*Liniment of Mustard.*

Etherial ext. mezereon,	.	.	.	.	40. gr.
Camphor,	.	.	.	.	120 gr.

Are dissolved in—

Rect. spirit,	.	.	.	.	4 fl. oz.
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To which are afterwards added—

Oil of mustard,	.	.	.	.	1 fl. drm.
Castor oil,	.	.	.	.	5 fl. oz.

The solubility of castor oil in spirit is here made use of, to enable an alcoholic liniment to retain the smoothness and lubricating qualities which a fixed oil imparts.

*Turpentine Liniment*

Contains—

Soft soap,	.	.	.	.	2 oz.
Camphor,	.	.	.	.	1 oz.
Oil turpentine,	.	.	.	.	16 fl. oz.

*Acetic Turpentine Liniment.*

Contains—

Oil of turpentine,  
Acetic acid,  
Lin. of camphor, in equal measures.

The latter, it must be remembered, is the oily, not the ammoniacal liniment.

### EXAMINATION QUESTIONS.

What is meant by over proof spirit, and how is it reduced to proof ?

What is meant by under proof spirit, and how can it be raised to proof ?

What is the difference between the strengths of the aromatic spirits and essences of the Pharmacopœia ?

What is the general process for making tinctures ?

Name the most notable exceptions to the general rule of the strength of tinctures ?

What is the difference between the alcoholic value of wines and tinctures ?

What condition promotes the permanence of ipecacuanha wine ?

### RECAPITULATION.

To reduce our spirit to proof, add to 100 measures of it as much water, as makes it measure 100, plus the number it was over proof.

Spirit under proof indicates spirit of such a strength as could be obtained by mixing proof spirit and water ; the number under proof indicates the number of measures of water in 100 measures, the remainder being proof spirit.

The aromatic spirits contain 2 per cent. of the essential oil giving them their name, the essences contain 20 per cent.—both by measure.

The general rule for the strength of tinctures, is that 1 fluid ounce contains the soluble matter from 1 drachm of the drug from which they are named. There are several important exceptions to this rule, notably, tincture of opium and tincture of quinine.

Medicinal wines resemble tinctures, but are much less alcoholic, proof spirit tincture containing nearly 50 per cent. of alcohol, and the wines from 12 per cent. to 18 per cent.

Ipecacuanha wine is more permanent if free acid be present.

## LECTURE XVI.

### OFFICIAL PRODUCTS OF DISTILLATION AND SUBLIMATION.

Hydrocyanic Acid—Nitric Acid—Ether—Aromatic Waters—Chloroform—  
Essential Oils—Oils of Lemon, Nutmeg, Mustard, Spirit of Nitre, Spirit  
of Ammonia—Official products of Sublimation—Chlorides of Mercury.

I PROPOSE to treat first of a group of pharmaceutical products obtained by distillation, containing a variety of diverse materials associated mainly by their mode of production, but also more or less by certain qualities in common. They are all liquids free from sediment and leaving no fixed residue, the appearance of sediment or fixed residue being evidence of faulty manipulation. The general features of the process having been described in a former lecture, I will now direct your attention more particularly to a few considerations connected with the products themselves, and only point out a few particulars relating to the process where modifications or peculiarities seem to require notice.

#### *Hydrocyanic Acid.*

Hydrocyanic acid is obtained by distilling yellow prussiate of potass with sulphuric acid and water. The yellow prussiate is dissolved in a portion of the water; the acid diluted with another portion and cooled; they are then mixed and slowly distilled, the products being carried into a receiver containing a third portion of the water. The water in the receiver serves to condense any gaseous hydrocyanic acid which may not be condensed with the water which distills; it may consequently be regarded as a case of distillation com-

bined with the solution of a gas. Fig. 16, page 96, represents an arrangement of apparatus which may be used for this purpose, substituting a sand bath for a water bath, and taking care that the tube of the condenser dips into the water contained in the bottle.

However carefully the process may be conducted, it is always desirable to control the accuracy by a volumetric examination of the distillate, and add the quantity of water which may thus be found necessary to reduce the strength down to precisely 2 per cent. As it also loses strength by keeping, old samples should not be used unless proved by analysis to have approximately the standard strength.

#### *Nitric Acid.*

The strength and purity are the only two points to which we need at present give our attention. The specific gravity is 1.42, which is advantageously fixed upon as being the most permanent, on account of stronger acids being reduced to this strength by boiling, and weaker acids increasing in strength by the same treatment up to the same point.

#### *Ether.*

Ether may be distilled in an arrangement similar to that described for hydrocyanic acid, provided there is an abundant flow of cold water to keep the condenser and receiver thoroughly cooled, and a safety funnel added to the flask; so that more spirit can be kept flowing in as the ether distills off. The contents of the retort are to be kept of about constant bulk. The ether is accompanied in the receiver by the water formed by the decomposition of the alcohol, and by a portion of alcohol which has escaped decomposition.

In a careful experiment, Mitscherlich obtained from every 100 parts of absolute alcohol,—

- 65 of ether,
- 17 of water, and
- 18 of undecomposed alcohol, in the receiver.

The precise nature of the changes which take place during the conversion of alcohol into ether and water, are not clearly established, and their study belongs rather to chemistry than pharmacy. I will therefore not enter into the question, beyond reminding you that alcohol contains the elements of ether and water, and that distillation with sulphuric acid converts the former into the latter, without itself undergoing any change, the name "sulphuric ether" being still frequently though incorrectly used. The product of distillation I have just said contains alcohol and water dissolved in the ether. The addition of a large bulk of water causes the precipitation of the greater part of both the alcohol and the water; the comparatively pure ether floating on the top may be decanted and further purified by a second distillation, which should be stopped when the total distillate has a specific gravity of 735 at 60° Fahr., as indicated by a specific gravity bead placed in the receiver. To make *pure ether* of the Pharmacopœia, a second washing is required to remove a portion of spirit still adhering, and another rectification from lime and chloride of calcium to remove the last traces of water.

#### *Aromatic Waters.*

The aromatic waters constitute a natural group of pharmaceutical products, the most of them being of very simple nature, and requiring little else than a few general remarks upon the class.

If we regard camphor as a concrete essential oil, we may say the waters are obtained by agitating the oil with water, by distilling the oil with water, or by distilling the material yielding the oil with water, to which we may add the non-official processes of rubbing the oil with sand or magnesia, agitating this with the water, and filtering; and lastly, dissolving the oil in spirit, and adding this to the water. Much has been said upon the relative merits of these various processes. We have not time to enter fully into their consideration now; but I must remark in passing, that the presence of spirit in the

waters is quite objectionable. I have repeatedly seen waters prepared by the aid of spirit turn sour by keeping, the spirit, no doubt, being converted into acetic acid. The use of magnesia has been objected to on account of its power of forming salts with the oils, part of the magnesia itself remaining in solution, and retaining more than the legitimate quantity of oil. As far as my experience goes, when it is necessary to extemporise a water, nothing is gained by the use of magnesia or sand over simply adding the oil to the water, hot in preference, using violent agitation, and filtering through paper.

A further objection to the use of magnesia is the chance of its interfering with any salt which may be prescribed along with the aromatic water; the chance of this is the greater in consequence of its solubility being increased by some at least of the essential oils.

In the Pharmacopœia camphor water is the only preparation of this class in which distillation is dispensed with.

Peppermint and spearmint waters are the only two in which distillation from the essential oil is directed—the flavour of the water prepared from the oil being usually preferred to that made directly from the herb, the keeping qualities of the former also being superior.

The others are all prepared by distilling the plant yielding the oil with water. The fruit of caraway, dill, fennel, &c., being rich in essential oil, 1 lb. is sufficient to yield a gallon of water. The flowers of rose and elder containing but a small percentage, 10 lb. are required to produce 1 gallon; in any case, the vegetable is placed in 2 gallons of water when 1 gallon is required to be drawn over, the object of the second gallon being to prevent scorching of the material in the still.

The waters, as a class, are simple aromatics, commonly used as vehicles for more potent remedies, imparting to them an agreeable flavour and warm carminative qualities; but there is one notable exception. Aqua laurocerasi is a powerful poison from the quantity, and the *uncertain* quantity, of hydrocyanic acid it contains. While the other waters may be taken

*ad libitum*, laurel water is given in doses of 5 to 30 drops. It is a pity that an article of this kind should be rendered official without there being any rule for insuring uniformity of its strength.

### *Chloroform.*

One part of rectified spirit and 16 parts of water are mixed in a large glass retort, and heated to 100° Fahr. Six parts of chlorinated lime and 3 parts of hydrate of lime are then added, and heat applied till active ebullition has commenced; the heat is then immediately withdrawn, and the distillation allowed to go on. It is necessary to have the still body much larger than the measure of the materials put into it, as considerable frothing takes place. It is also necessary to attach a thoroughly efficient condenser, supplied with abundance of cold water, to avoid the loss of so volatile a product as chloroform, which boils at 142° Fahr. When once in the receiver, however, it is in some measure protected from loss by evaporation. Its specific gravity being 1.49, it sinks to the bottom of the water that distills over with it. In the crude state, it is contaminated with spirit which has escaped decomposition, and a trace of essential oil, resulting from the reaction. It is freed from the former by washing with water; and from the latter by agitation with oil of vitriol, which decomposes the oil, but does not act upon the chloroform. It is decanted from the oil of vitriol, and finally purified by agitation with slaked lime and chloride of calcium, which combine with adhering water, and fix it while the chloroform is again distilled.

It is important to use pure sulphuric acid in the latter part of the process, as a contamination of nitric acid gives the chloroform a tendency to decompose by keeping, more especially if exposed to light.

### *Essential Oils.*

The essential oils are distilled in the same manner as the aromatic waters, except that the quantity of water is dimin-



ished ; and sometimes the water is returned to the still again and again with more of the herb yielding oil. The oil accumulates in such quantity that it may be readily skimmed off the surface or separated by a Florentine receiver, which is a vessel shaped somewhat like a coffee-pot, with a short spout on its rim and a long spout attached to the bottom.

The whole products of distillation flowing into the Florentine receiver, they separate by subsidence, the water flowing off by the long spout, which is so placed as to carry away the fluid which occupies the bottom of the receiver, at the same time that any supernatant oil is flowing away in an opposite direction by the short spout.

Many of the essential oils are directed by the Pharmacopœia to be distilled in Britain ; not that it is impossible to distil good oils abroad, nor impossible to have inferior oils of British manufacture, but that the home distilled oils are, as a rule, of a finer quality than those produced abroad.

The oils of dill, chamomile, caraway, and cloves, are directed to be prepared in Britain ; the oil of aniseed in Europe. Cajeput is defined as a foreign oil ; and in regard to the oils of cinnamon and cloves, we are left at liberty to derive them from any source.

Oil of lemons, though an essential oil, may be obtained either by expression or distillation.

Oil of nutmeg obtained by expression contains both fixed and volatile oil ; that obtained by distillation contains, of course, the volatile oil only. Thus, the case of nutmegs contrasts with that of almonds and mustard, inasmuch as the two latter seeds contain the fixed oil already formed, but only give rise to their essential oil by decomposition, which takes place on the contact of water. The nutmegs contain both fixed and volatile ready formed, consequently yield them both when subjected to pressure.

Most of the essential oils are lighter than water, but there are a few exceptions, *e.g.*, the oils of cassia, cinnamon, and cloves being heavier.

Oil of mustard is perhaps the only oil which requires a special notice at present. If the mustard seed be pressed dry it yields a sweet fixed oil, in which there is no trace of the pungency of mustard; but if the mustard be macerated in water and distilled, an oil comes over which is characterised by extreme pungency and irritating action upon the skin. The essential oil thus obtained does not exist ready formed in the seed, but is the product of a decomposition which takes place when two of the constituents come in contact with water. Myronic acid, by the action of synaptase, yields the oil of mustard.

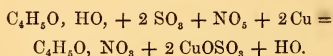
The essential oil of bitter almonds is another instance of an oil being produced by the action of water upon principles contained in the material from which it is distilled; it, however, is not an official product.

*Spiritus Etheris Nitrosi.*

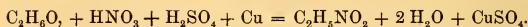
Nitric acid,	.	.	3 fl. ounces.
Sulphuric acid,	.	.	2 do.
Copper wire,	.	.	2 ounces.
Rect. spirit,	.	.	3 pints, or a sufficient quantity.

The sulphuric acid being added to 1 pint of spirit, and then  $2\frac{1}{2}$  oz. nitric acid added, the mixture is poured upon the copper wire in a still fitted with a thermometer. Heat being applied to keep the temperature between  $170^{\circ}$  and  $180^{\circ}$  Fahr., nitrous ether distills along with some unchanged spirit. The change which takes place may be represented as follows:—

*Old Notation.*



*New Notation.*



The nitrous ether and spirit which pass over require a thoroughly efficient condenser to reduce them to the liquid state, as the nitrous ether boils at  $61.5^{\circ}$  Fahr.

The remaining half ounce of nitric acid is added when the action grows slack, and distillation is continued till 15 fl. ounces are obtained. To the distillate is added as much spirit as will bring the density to  $\cdot 845$ ; the density of nitrous ether being  $\cdot 947$ , and that of rectified spirit being  $\cdot 838$ . It will be observed that the sp. gr. is no guide to the medicinal value of the product, as its density may be increased by either the addition of water or of nitrous ether.

When new it is nearly free from acid, and will on being mixed with twice its volume of saturated solution of chloride of calcium yield 2 per cent. of nitrous ether by settling. The nitrous ether being partly retained in solution, the 2 per cent. does not show the whole quantity present, but the quantity in excess of what is retained by the solution of chloride of calcium and spirit from which it has separated. The total quantity of nitrous ether present should be 10 per cent., of which 8 per cent. remains dissolved and 2 rise to the surface.\*

We are indebted to Professor Redwood for this process, which yields a product of greater and more uniform strength than that which was formerly followed. After trying various materials capable of evolving nitrous gas from nitric acid, he adopted copper as the most satisfactory. Copper being a costly material, and the nitrate formed being of very limited consumption, manufacturers have since substituted other substances with some pecuniary advantage.

Though it is not difficult to make spirit of nitre to possess the qualities indicated in the official tests, it is impracticable to obtain it thus in commerce. As sent out by the best makers it is generally found decidedly acid, and incapable of yielding the required film of nitrous ether, defects no doubt resulting from the instability of the ether. It has been stated that the acidity is due to acetic acid, but the powerful action it commonly exerts upon iodide of potassium would rather indicate the liberation of some nitrous compound.

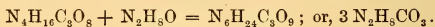
\* For further particulars, see Attfield's "Chemistry," page 395, &c.

*Spiritus Ammonice Aromaticus.*

When the ordinary carbonate of ammonia is added to the spirit, water, and liquor of ammonia used in preparing this article, an additional equivalent of ammonia enters into combination, forming the salt known as monocarbonate of ammonia. The change may be expressed thus—

*Old Notation.*

Or, according to the new equivalents—



The salt thus formed being much more volatile than the ordinary carbonate of ammonia, distills along with the spirit and oils of lemon and nutmeg. Cloves were formerly used in this preparation, but they are now omitted, as they cause the spirit to become brown by keeping.

*Sp. Ammon. Fetidus*

Contains caustic instead of carbonate of ammonia, and is impregnated with the essential oil of assafœtida.

*The Products of Sublimation.*

The various substances thus obtained are usually manufactured on the large scale, and are consequently rather subjects of study under chemistry and materia medica than pharmacy: the only two for which the editors of the Pharmacopœia have thought it necessary to provide formulæ are the two chlorides of mercury. The chief articles of materia medica obtained by sublimation are—acid, benzoic; acid, arsenious; ammonia carbonate; ammonium chloride; camphor; mercury perchloride; mercury subchloride; iodine; sulphur.

They should all be entirely volatilised by heat, and some are also volatilised slowly at ordinary atmospheric temperatures.

Benzoic acid, carbonate of ammonia, camphor, and iodine belong to the latter section.

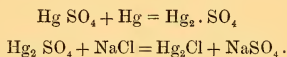
Carbonate of ammonia is decomposed by exposure to the air into a very volatile carbonate, which evaporates, and a more fixed carbonate, which is only slowly volatilised, except at a higher temperature.

*Perchloride of Mercury,*

Obtained by subliming a mixture of persulphate of mercury with chloride of sodium. In the Pharmacopœia formula a small portion of black oxide of manganese is added to the mixture, with the object of insuring the absence of subsalts from any imperfectly oxidised mercury present in the sulphate.

*Subchloride of Mercury.*

The same materials are used in this as in the last, with the addition of metallic mercury, which is first rubbed with the sulphate of mercury to reduce it from a persulphate to sub-sulphate.



The process having been described in the lecture on sublimation, we need now only draw attention again to the fact that it undergoes partial decomposition on sublimation, a small quantity of corrosive sublimate and metallic mercury being formed.

QUESTIONS FOR EXAMINATION.

What is the characteristic quality of all products of sublimation, and how would you divide them into two sub-classes?

What are the distinctive characters of products of distillation?

What circumstances render it important to have hydro-cyanic acid constantly tested?

What are the usual doses of aromatic waters? Give a

notable exception. State some objections to the use of spirit or magnesia in extemporising an aromatic water.

State the general mode of procuring essential oils, and note one or two exceptional cases.

What characters should spirit of nitrous ether possess? What defects are commonly found in commercial samples? What is the value of testing its density?

#### RECAPITULATION.

Products of sublimation should be entirely volatile, some at ordinary atmospheric temperatures, others only at higher heats.

Products of distillation should be free from sediment and entirely volatile also.

Hydrocyanic acid, being very liable to variation, requires systematic testing.

The aromatic waters should contain nothing but water and essential oil; if extemporised, the use of magnesia, or spirit, is objectionable; with the exception of laurel water, they have little activity.

Essential oils usually exist in the plants from which they are obtained, and are extracted by distillation; oil of lemons is an exception, inasmuch as it is extracted by expression; oil of mustard is an exception, in being formed by the decomposition of other principles contained in the seeds.

Spirit of nitrous ether should contain but little free acid, and should evolve a film of nitrous ether on mixing with solution of chloride of calcium; it is not difficult to make it with these qualities, but impossible to keep it so. Its density does not indicate its strength.

## LECTURE XVII.

### OFFICIAL PRODUCTS OF FUSION.

Ointments—General Instructions—Mercurial Ointment—Spermaceti Ointment—Stirring—Nitrate of Mercury Ointment—Plasters—Mercury, Belladonna, Cantharides, &c.—Other Products of Fusion—Purified Bismuth—Bromide and Iodide of Potassium.

#### OINTMENTS, PLASTERS, &c.

MANY of the official ointments do not involve fusion, but they are most conveniently treated along with others, which it is necessary to reduce to the fluid state in the process of making.

Those which are not melted are—

Ung. aconitiæ,	. . . . .	8 gr.	to 1 oz.
antim. tart.,	. . . . .	$\frac{1}{4}$ oz.	„
atropiæ,	. . . . .	8 gr.	„
belladon,	. . . . .	80 gr. extract	„
cadmii iodidi,	. . . . .	62 gr.	„
creasoti,	. . . . .	1 drachm	„
gallæ,	. . . . .	80 gr.	„
gallæ cum opio,	. . . . .	32 gr. opium	„
hydrarg.,	. . . . .	1 part in 2.	
ammoniati,	. . . . .	62 gr.	to 1 oz.
iodid. rub.,	. . . . .	16 gr.	„
subchlor.,	. . . . .	80 gr.	„
iodi,	. . . . .	32 gr.	„
plumbi acet.,	. . . . .	12 gr.	„
carb.,	. . . . .	62 gr.	„
iodidi,	. . . . .	62 gr.	„
potassæ sulphuratæ,	. . . . .	30 gr.	„
potassii iodidi,	. . . . .	64 gr.	„
sulphuris,	. . . . .	120 gr.	„
iodidi,	. . . . .	30 gr.	„
veratriæ,	. . . . .	8 gr.	„
zinci,	. . . . .	80 gr.	„



In making these, the chief point requiring attention is to have the dry, active materials finely powdered and thoroughly mixed. This requires special care in the case of tartarised antimony, iodide of cadmium, sulphurated potass, and iodide of sulphur, all of which are apt to be rough and unpleasant to use unless great attention be paid to the careful grinding. The iodide of sulphur ointment may be made smooth, and of a dark brown colour, by the addition of a little iodide of potassium and water, which would probably be an improvement to the official formula.

Sulphurated potass ointment is also more readily made smooth by the addition of a little water.

*Ung. Potassii Iodidi.*

In this instance the smoothness of the ointment is insured by the salts being dissolved in water. The addition of carbonate of potass to this ointment in the present Pharmacopœia prevents its becoming yellow by keeping, which was apt to be the case with the old form, the ointment becoming rancid and yellow from the liberation of iodine. The addition of hyposulphite or sulphite of soda has been suggested to prevent this ointment becoming discoloured.\* Whether it would have the effect of preventing rancidity or not, it would no doubt prevent the liberation of iodine, which usually accompanies this change.

*Ung. Belladonnæ.*

The quantity of water which is added to this ointment should be just so much as to reduce the extract to a soft condition. As the extract of commerce is rarely of a pilular consistence, it requires very little water. If too much is added, the mixing is less perfect. An excess of water probably increases the tendency to mouldiness. The addition of a little oil of cloves would tend to its preservation, but as the formula stands, it is better prepared fresh when required. Probably the use of

\* Pharm. Jour. Nov. 11, 1871.

spirit or glycerine for softening the extract would diminish its tendency to mouldiness.

*Ung. Gallæ.*

We are not directed to use heat to soften the lard in the preparation of ointments which contain simply lard and a dry powder; but in cold weather the use of a little heat is desirable, as it very much facilitates the production of a perfect mixture. When we have recourse to artificial heat with such an object, it is best to use just so much as reduces the lard to a creamy consistence, as we thus avoid the chance of the powdered material sinking to the bottom, and save both the time which would have been occupied in rendering it fluid, and that which would have been spent in stirring it till cold again. In the case of ung. gallæ, there is an additional reason for avoiding much heat, as the galls run into hard knots if heated too much; after which it is almost impossible to make the ointment fit for use.

*Ung. Hydrargyri*

Requires long-continued rubbing at a gentle heat, just sufficient to keep the ingredients in a creamy condition. It is usually made on the large scale by machinery. As the mercury is only oxidised to a very small extent, it may be recovered in the metallic state by melting the ointment and allowing it to settle; if then washed with a little ether to remove the adhering grease, 33 parts of the ointment should yield 16 of mercury.

It is desirable that testing of purchased samples should not be entirely omitted, as the appearance is not much guide to its quality. A simpler test than the above, though not a crucial one, is the taking of its specific gravity. One hundred parts by weight of mercurial ointment consist of—

Mercury,	.	48·5 (sp. gr. 13·5)	= 3·5 measures.
Grease,	.	51·5 (sp. gr. about '9)	= 57·2 „
		<hr/>	
Total,	.		60·7

Consequently 100 ounces by weight should measure 60·7, or, striking off the fraction, say  $2\frac{1}{2}$  oz. by weight equals  $1\frac{1}{2}$  oz. by measure. In making an examination of this kind, it is most important to avoid any air-bubbles being entangled in the ointment, which would add to its bulk without increasing its weight, and thus imply a deficiency of strength.

The pale colour of a sample which recently fell under my notice led me to examine its specific gravity, which turned out to be about 1·6, or nearly what the above calculation would make it—*i.e.*, 1·65; and the paleness of its colour proved to be the result of a less perfect division of the mercury.

The official instruction had been obeyed, literally obeyed, for metallic globules were not visible to the naked eye; but in many cases the trituration is continued till the mercury ceases to be visible with the aid of a lens of one or two inches focus. It would probably be better that this latter degree of division should be officially required.

#### *Ung. Veratricæ.*

A little oil is added in the preparation of this ointment to enable the veratria to be rubbed quite smooth without dispersing any of it in dust, which is exceedingly irritating to the eyes and nose of the operator.

The bases of the remaining ointments require to be melted as the first step in their making.

#### *Ung. Cetacei—Ung. Simplex.*

These may be taken as typical examples; in the former some latitude is allowed as regards the quantity of oil to be used, so that the ointment may be firmer or softer according to the desire of the consumer, or the requirements of the season. The condition of the ointment is also influenced by the stirring. The Pharmacopœia says, stir constantly while it cools. But it is not sufficient to stir it till of a creamy consistence; the solid ingredients are more or less crystallisable, and as the cooling continues they deposit partly in an indistinctly

crystallised form, which gives a hardness or rigidity to the ointment which is very objectionable ; and it is difficult afterwards to make it smooth by rubbing ; but if the stirring be continued constantly till the ointment is so cold that no more crystallisation will take place, the crystals which are formed are very small and not agglomerated, so that the ointment is soft and plastic instead of being rigid. An ointment which contains so small a proportion of oil that it is firm while in this plastic state, is less affected by changes of temperature than an ointment which has the same degree of firmness, with more oil, and having been left at rest during the latter part of the cooling.

A sample of spermaceti ointment containing—

Spermaceti,	.	.	.	5 oz.
White wax,	.	.	.	3 oz.
Almond oil,	.	.	.	1 lb.

melted and stirred till it was in a semi-fluid semi-solid state at 93° Fahr., a portion taken out and cooled at rest was hard at 63°; but another portion having been stirred till it fell to 73°, was plastic at 63°. It would bear the ordinary changes of season without becoming either too hard or too soft for convenience, but if the quantity of oil had been so much increased that the ointment was of a conveniently soft consistence in cold weather with the small degree of stirring, viz., only till the ointment was in a creamy semi-solid condition, then any rise in the temperature would make it unmanageably soft. Different samples of oil appear to me to require different proportions of the hard materials to give them a suitable consistence.

Though the consistence of spermaceti ointment is left indefinite, it is intended to be softer than the simple ointment which contains—

Lard,	.	.	.	3 oz.
White wax,	.	.	.	2 oz.
Oil of almonds,	.	.	.	3 oz.

*Ung. Cantharidis.*

This is prepared by infusing cantharides in oil, filtering and adding melted wax to give the required consistence. The ointment is of a light-yellow colour, and should be free from particles of the fly.

*Ung. Elemi—Ung. Resinæ.*

These two ointments require filtering while in the fluid state, as some clotty matter separates on the mixture of the resin with the grease; this is the case even if the resin and the grease are both quite clear before mixture.

The ung. resinæ of the British Pharmacopœia is much harder than usually liked. The consistence I find generally preferred is obtained by using the resin and simple ointment in the proportion ordered but omitting the wax—

Resin,	.	.	.	8 oz.
(Yellow wax,	.	.	.	4 oz.)
Simple ointment,	.	.	.	16 oz.

*Ung. Hydr. Compositum*

Is half the strength of the simple mercurial ointment, and has  $\frac{1}{8}$ th of its weight of camphor added. Camphor is usually powdered by rubbing it with a few drops of spirit, but in a case like this a little of the olive oil which forms one of the ingredients of the ointment may be used. The addition of camphor makes the ointment much softer than it would be without it—a circumstance which is easily understood if we keep in mind that the camphor is a concrete essential oil which becomes fluid by union with the grease.

*Ung. Hydr. Nitratis.*

There has been much said about the difficulty of making a good nitrate of mercury ointment, and a great number of formulas have been suggested. There need not, however, be any deviation either from the ingredients or the modes of operat-

ing directed in the British Pharmacopœia. It is essential that the solution of nitrate of mercury be mixed with the grease while they are so warm as to cause a brisk effervescence. If the heat be too great, there is fear of loss from the ointment boiling over the top of the pan, and if the heat be insufficient, more is to be applied till free effervescence takes place. If long kept, the ointment becomes dark coloured, the evaporation of nitric acid being one change, but perhaps not the only change which it has thus undergone. It is better, therefore, to make only so much at a time as will be consumed before change of colour takes place. It is necessary to avoid bringing this ointment into contact with metallic substances, which might decompose the nitrate of mercury it contains, consequently the Pharmacopœia directs it to be made in a porcelain vessel. After the ointment is completed it is not very rapidly decomposed by either copper or iron.

In the British Pharmacopœia formula there are 15 oz. of lard to 32 of olive oil, which makes the ointment rather soft, but if these quantities be reversed,—that is, 32 of lard to 15 of oil,—a more convenient consistence is produced.

*Ung. Plumbi Subacetatis Comp.*

Solution of subacetate of lead,	.	.	.	6 fl. oz.
Camphor,	.	.	.	60 gr.
White wax,	.	.	.	8 oz.
Oil of almonds,	.	.	.	1 pint.

The wax is to be melted with four-fifths of the oil, and the camphor to be dissolved in the remaining fifth, that it may be kept separate to avoid the loss of camphor which would take place if the whole were heated together. When the wax and oil cool so much as to become milky, the solution of acetate of lead is stirred in, forming a mixture analogous to an emulsion.

The subacetate of lead readily emulsifies with oil, having much less tendency to subside than most aqueous solutions.

*Unguentum Sabinæ.*

Fresh savin tops are bruised and digested in the melted grease for twenty minutes, by which they impart to it some resin and essential oil, upon which the value of the ointment depends, and some chlorophyl, which gives it its usual green colour. As the green colour is rather fugaceous, the brightness may be considered an indication of the ointment being fresh.

*Emplastra.*

The plasters bear a general resemblance to the ointments in their chemical nature, their mode of manufacture, and their use. They are all prepared with the aid of heat, and all liable, though in various degrees, to be injured by over heating. In several instances water is added, which prevents the temperature rising above boiling water heat, the water being driven off during the process.

*Emplastrum Ammoniaci cum Hydrargyro.*

Ammoniacum,	.	.	.	.	12 oz.
Mercury,	.	.	.	.	3 oz.
Olive oil,	.	.	.	.	1 fl. dr.
Sulphur,	.	.	.	.	8 gr.

The sulphur is dissolved in the oil by the aid of heat, and with this the mercury is triturated till globules are no longer visible; then the ammoniac previously melted is added, and the whole mixed.

It seems remarkable that 3 oz. of mercury can be "killed" by trituration with so small a quantity as a fluid drachm of oil and 8 grains of sulphur.

The remarkable power of a small quantity of sulphur to "kill" a large quantity of mercury is well known to those who extract gold from quartz by amalgamation, the presence of a little sulphur causing a great loss of mercury, from the surface of the globules becoming tarnished, so that they lose



their power of agglomerating. The same action takes place in this process, the bulk of the mercury remaining in the metallic state, only a very small portion being converted into sulphide, in the form of tarnish on the surface of the globules, which facilitates their division and prevents them from re-uniting.

*Emplastrum Belladonnæ.*

Belladonna plaster was formerly made by mixing the ordinary watery extract with resin or soap plasters, but by this mode an imperfect compound was formed which was deficient in adhesiveness.

In the present mode of preparing belladonna plaster the ordinary aqueous extract is rubbed with about twice its weight of spirit, which dissolves the resinous and active part and leaves the mucilaginous matters, which are worthless and do not combine well with the resin plaster; by this process the resulting compound is more adhesive and more fusible. As this plaster is readily injured by heat, it is prepared in a water bath, and requires care in spreading, to have the iron as gently heated as it is practicable to operate with.

It has been stated that a plaster of still better quality is made from an alcoholic extract of belladonna root.

*Emplastrum Calefaciens.*

Four ounces of powdered cantharides is macerated in boiling water for six hours, the liquor pressed off and evaporated to one-third, and added to—

Exp. oil nutmegs,	}					
Yellow wax,						
Resin,						
Soap plaster,		.	.	.	.	3½ lb.
Resin plaster,		.	.	.	.	2 lb.

From the absence of the powder of cantharides, and the presence of some water, the colour of this plaster is creamy yellow.

It is customary to pour the melted plaster into water, and

before it hardens to pull the mass out, double it and pull it again, by which treatment it acquires a silky appearance, a brighter colour and greater opacity than it would have if simply cast into moulds. After this pulling process it is weighed into half-pound pieces and rolled upon a wet stone slab, to form rolls of 8 or 10 inches in length.

*Emplast. Cantharidis*

Contains one part of cantharides in three. It is apt to be very crumbly, unless beaten till cold. The instructions to stir the mixture while it cools are insufficient to insure the production of a plastic plaster. Before it is cool it becomes too hard to stir, and as soon as this occurs it is better to transfer it to a large mortar and beat it till cold. It may be made more plastic by increasing the quantity of resin and grease, and using little or no wax. Care must be taken, however, not to make the plaster more adhesive than enough, as it would give much pain to remove an adhesive plaster from a blistered surface.

*Emplastrum Opii.*

The present opium plaster contains one part of powdered opium and nine of resin plaster. Its predecessor in the London Pharmacopœia contained extract of opium softened with water, added to the melted plaster and evaporated to a suitable consistence. Probably an alcoholic extract of opium would make a better plaster than either.

*Emplastrum Plumbi.*

This plaster is a salt, in which the oxide of lead is the base; and the oil supplies the fatty acids. The oil being a combination of glycerine with oleic and other acids, undergoes decomposition by boiling in contact with oxide of lead and water. The glycerine separates and is removed along with the water which has not evaporated during the process; the heat of a steam bath is recommended, as a higher heat long continued causes

further decomposition of the oil, and even reduction of the lead to the metallic state.

*Emplastrum Resinæ—Emplastrum Saponis.*

Both these contain lead plaster, resin, and soap, the one or the other addition preponderating as the name indicates. They are more plastic and more adhesive than the lead plaster.

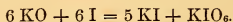
There are various other processes of fusion in the Pharmacopœia. In the preparation of some salts, and the purification of sulphuret of antimony and metallic bismuth, the latter is perhaps the most interesting of the groups, and has been the subject of much criticism. Commercial bismuth is usually contaminated with various metals which are more or less readily oxidised by the contact of fused nitrate of potass, by which arsenic, its most frequent and most important impurity, is removed. If any considerable percentage of copper is present it is but imperfectly separated by this treatment, but its presence is guarded against by the testing with ammonia which is enjoined.

The process answers satisfactorily in the absence of copper, and any samples of commercial bismuth which have a considerable contamination of this metal should not be used for this purpose; they may, however, be purified by being dissolved in nitric acid, and precipitated by dilution with water as in the official process for bismuthi subnitratis, the copper remains in solution; and by collecting, drying, and igniting the precipitate with charcoal it is again reduced to the metallic state; any foreign metals, if present, being left in the nitric acid solution, the reduced bismuth is now pure, excepting as regards arsenic. If both arsenic and copper are present in the original sample, it is desirable to use both processes of purification.

The processes for bromide and iodide of potassium involve the fusion with charcoal to reduce any oxysalt which might otherwise escape decomposition if heat alone were used.

The decompositions may be briefly shown thus:—

Six equivalents each of iodine and caustic potass unite to form five equivalents of iodide and one of iodate.



By fusion the iodate is converted into iodide with the liberation of its oxygen, but a small quantity is apt to escape decomposition unless the charcoal be added, which effectually abstracts the oxygen and flies off as carbonic acid or carbonic oxide.

A precisely similar change takes place in the case of the bromide. As the decomposition takes place with some violence, it is effected by throwing the mixed salts and charcoal in small successive portions into a red-hot crucible, thus each portion becomes deflagrated before the addition of the next. The reduction is complete when the whole mass has been brought into a state of quiet fusion. The salt is then poured out, and when cold dissolved in water and filtered to remove excess of charcoal, and the clear solution evaporated and crystallised.

#### EXAMINATION QUESTIONS.

When solid substances are to be mixed with grease for the production of ointment, what modes of insuring smoothness may be adopted ?

Describe two methods of testing the strength of mercurial ointment, and state their relative merits.

In making spermaceti ointment, how long should the stirring be continued ?

Why is cantharides plaster made nearly destitute of adhesiveness ?

What common impurity of metallic bismuth is not satisfactorily removed by fusion with nitrate of potass ? and by what method may the bismuth be freed from it ?

## RECAPITULATION.

In making ointments the active matter should be smooth, and uniformly mixed. The smoothness is usually insured by careful grinding, at other times by solution.

The strength of mercurial ointment should be tested; most advantageously by separating and weighing the mercury, or more readily though less accurately by taking its sp. gr.

The stirring of spermaceti ointment should be continued till its temperature falls to about 70° Fahr., to insure a plastic and smooth condition.

Cantharides plaster should possess little adhesiveness, lest, in removing it after use, it tear the skin off the blistered surface.

Fusion of bismuth with nitrate of potass is recommended to purify it from arsenic. Copper is more effectually removed by solution in nitric acid, precipitation with water, and fusion with charcoal.

## LECTURE XVIII.

### OFFICIAL SALINE PREPARATIONS, ETC., CRYSTALLISED, PRECIPITATED, SCALED, OR GRANULATED.

Crystallised Salts having Volatile Acid or Base—Precipitates, Oxide of Antimony, Subnitrate of Bismuth—Chloride of Calcium, &c.—Separation of Iron—White Precipitate—Carbonate of Magnesia—Granulated Sulphate of Iron—Carbonate and Citrate of Potass—Citro-Tartarate of Soda—Scaled Salts, Sulphate of Beberine, Citrate of Iron and Ammonia, &c.

THIS is a considerable group of substances, including, besides salts, some metallic oxides, sulphides, &c. It will only be possible to take a very brief view of them, but this is of less consequence, as they are also treated by the lecturers on chemistry and materia medica, and the processes of crystallisation and precipitation in the abstract have already been treated in my former lectures.

We will first consider one or two cases of crystallisation in which some small additional precautions are required.

#### *Ammoniac Benzoas—Ammoniac Phosphas.*

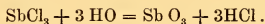
These are two instances of simple combination of the acid with the alkali; but the alkali being volatile and held by only a feeble affinity, it is desirable to keep it in excess, adding more towards the end of the process to compensate for what is lost by evaporation. Some salts of fixed bases with volatile acids, or of the volatile alkali with fixed acids, though sufficiently stable to bear exposure to the air in the dry state, are liable to part with a portion of their volatile constituent while their solutions are undergoing evaporation.

The loss of acid in this way is exemplified in the case of plumbi acetate, in which we are directed to add a little acetic acid at the end of the evaporation to compensate for that lost in the process, and thus ensure the absence of sub-acetate in the crystals.

The common instructions of the British Pharmacopœia to drain and dry the crystals on filtering paper, are of course only suitable for experimental operations. The centrifugal drainer which I mentioned in a former lecture may with the greatest advantage be used on the manufacturing scale.

*Antimonii Oxidum.*

When solution of chloride of antimony is diluted with a large quantity of water, a precipitate is thrown down, consisting of chloride and oxide of antimony united, the solution from which it has been thrown down still retaining a small quantity of antimony and a considerable quantity of hydrochloric acid, produced by the mutual decomposition of chloride of antimony and water—



The precise composition of the precipitate depends upon the quantity of water used, the composition varying from one equivalent of chloride with two of oxide to one of chloride with thirteen of oxide. The quantity of chloride present diminishes as the washing of the precipitate continues. The liquor of the chloride of antimony contains various impurities, chloride of iron most abundantly, and smaller quantities of copper, lead, and arsenic. These are washed away along with the hydrochloric acid; when they are sufficiently removed, the remaining undecomposed chloride of antimony contained in the precipitate is decomposed by the addition of carbonate of soda, carbonic acid being liberated, chloride of sodium and oxide of antimony formed. The precipitate is subjected to a second washing to remove the soda salts.



*Bismuthi Subnitrates.*

The process for the preparation of this salt is very similar to that which I have just described in connection with oxide of antimony. The metallic bismuth is first dissolved in nitric acid to form an acid nitrate  $\text{BiO}_3 \cdot 3 \text{NO}_5$ , which, on dilution with a large quantity of water, throws down the subnitrate  $\text{BiO}_3 \cdot \text{NO}_5$ , leaving in solution a strongly acid nitrate  $\text{BiO}_3 \cdot 9 \text{NO}_5$ , together with nitrates of copper, iron, or lead, if these metals were present in the bismuth, but arsenic, which is a frequent impurity of bismuth, is thrown down in combination with the subnitrate. It is, therefore, more especially important to have the bismuth freed from arsenic before it is used for this purpose.

It is said that by long continued washing the whole of the nitric acid may be washed out of this precipitate, leaving only the oxide; the Pharmacopœia, however, fixes the extent of washing which has to be used, and gives as the composition of the product, the formula  $\text{BiO}_3 \cdot \text{NO}_5 \cdot 2 \text{HO}$ , or  $\text{BiNO}_4 \cdot \text{H}_2\text{O}$ .

It is to be dried at a temperature not exceeding  $150^\circ$  Fahr. ( $= 65.5^\circ$  C.) to avoid the expulsion of the water of hydration which it contains.

The smooth, soapy feeling, white bismuth frequently met with in commerce is a basic chloride, or a combination of basic chloride and nitrate. It is preferred where a cosmetic is required, but should not be substituted for the official preparation in pharmacy.

*Carbonate of Bismuth*

Is precipitated by pouring the solution of the acid nitrate into solution of carbonate of ammonia. As there is not the same opportunity of washing out the nitrates of other metals, in this case it is the more important to use bismuth which is free from them.

*Oxide of Silver*

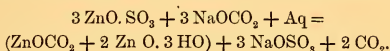
Is precipitated from the nitrate by means of lime water, which is preferred to the alkaline solutions, as it is less likely to be contaminated with carbonate, and thus avoids the chance of carbonate of silver being thrown down along with the oxide. To insure absence of carbonate, freshly prepared lime water should be used.

*Chloride of Calcium*

Is obtained by neutralising hydrochloric acid with carbonate of lime. Most of the native carbonates of lime contain oxide or carbonate of iron; and with the view of separating the protochloride of iron thus introduced a little chlorinated lime is added to the solution, by the action of which it becomes converted into perchloride, and is subsequently precipitated as peroxide by the addition of slaked lime. Proto-salts of iron are very imperfectly precipitated in this way, but persalts are completely separated, hence the use of the chlorinated lime. The same mode of separating iron is adopted in the preparations of zinc, using chlorine water and carbonate of zinc in place of chlorinated lime and slaked lime.

From the solutions thus purified the carbonates of lime and zinc may be precipitated by the addition of alkaline carbonates.

In precipitating carbonate of zinc, effervescence takes place from the precipitated carbonate of zinc containing only one equivalent of carbonic acid to three of oxide of zinc—

*Ferri Peroxidum Humidum—Ferri Peroxidum Hydratum.*

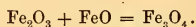
The humid or moist peroxide precipitated from persulphate of iron by caustic soda, and after being washed and drained is preserved without drying; but it should not be preserved long, as the only advantage it possesses over the hydrated oxide is its greater solubility, and this it in a great measure loses when kept for a length of time, even if preserved moist.

The hydrated oxide is prepared by drying this precipitate below  $212^{\circ}$ , by which it retains one equivalent of water and a considerable degree of solubility.

By a higher heat it loses the water of hydration, and with it its ready solubility.

### *Ferri Oxidum Magneticum.*

When peroxide and protoxide of iron are precipitated together they unite, forming the black magnetic oxide, thus—



The official process yields a precipitate containing an excess of peroxide of iron and some water of hydration. It is not probable there is any advantage in its use in medicine as it is decomposed into proto- and per-compounds by union with acids.

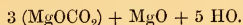
### *Hydrargyrum Ammoniatum.*

There is no peculiarity in the process for this compound, but the nature of the precipitate is such as does not usually belong to precipitates resulting from the decomposition of a metallic chloride with ammonia. In most cases chloride of ammonium would be formed, and the metal precipitated as an oxide, but in this case the precipitate has such a composition as would indicate its nature to be a chloride of ammonium in which two equivalents of hydrogen are replaced by one of mercury —  $\text{NH}_2\text{HgCl}$ —chloride of ammonium being also formed at the same time. Slight variations in the process give precipitates with different compositions and slightly different properties; they are all known as white precipitate of mercury. The official product is entirely volatilised on the application of heat, without first undergoing fusion. (See further in Attfield's "Chemistry," &c.)

### *Magnesiae Carbonas—Magnesiae Carbonas Levis.*

It must be noted that when carbonate of magnesia is ordered, the heavy is to be used unless the light is specially indicated.

They are of the same composition, viz., three equivalents of neutral carbonate with one equivalent of hydrate and four of water, or—



The physical condition depends upon the degree of concentration of the solutions from which the precipitate is thrown down, and some other minutiae which manufacturers keep to themselves.

The precise processes of the British Pharmacopœia are probably rarely followed, but as the result is obtained satisfactorily by other methods this is of no moment.

### *Ferri Sulph. Granulata.*

This is a precipitate formed by adding a strong aqueous solution of sulphate of iron to spirit of wine. The salt is precipitated as minute crystals of precisely the same composition as the salt crystallised in the usual way.

It has been said to be a refinement without an advantage; but granulation as directed in the Pharmacopœia is a convenient mode of obtaining the photosulphate of iron free from the persulphate. The former salt being insoluble in spirit is precipitated, while the latter, from its free solubility in spirit, remains in solution; the degree of purity thus obtained is greater than the salt crystallised in the usual way, unless the process was conducted with more than ordinary care, and the granulated salt is found to be less prone to oxidation by exposure to the air.

Some manufacturers produce sulphate of iron in large crystals with this same property—a tendency to effloresce without oxidising, but by what means they obtain these results I am not informed.

### *Potassæ Carbonas—Potassæ Citras.*

These two salts are granulated by evaporating the solutions to dryness with constant stirring, by which operation an indistinct crystallisation takes place; but as there is no mother

liquor separated, the salt is not purified by the process, and to obtain satisfactory products the chemicals operated with must be sufficiently pure before granulation is commenced. In speaking of the phenomena of solution, I pointed out that a saturated solution of carbonate of potass does not dissolve any appreciable quantity of sulphate. We consequently have the means of purifying the carbonate of potass, known in commerce as "pearl ash," by simply dissolving out the carbonate in the smallest possible quantity of water, by which the sulphate is left behind, and the solution obtained is sufficiently pure to be granulated for medicinal use. The usual process of crystallisation is not used for these salts, as they are deliquescent, and the drainage and drying of the crystals would be troublesome.

*Sodæ Citro-Tartras. Eff.*

The granulation of this salt, or rather mixture of salts and acids, is effected by using just so much heat as causes incipient semi-fusion, stirring till the powder begins to agglomerate, and then withdrawing the heat that further decomposition may not take place. The salt is then passed through a coarse sieve to reduce any lumps which might be too large, and then the fine dust is sifted out with a fine sieve; the dust being worked up with the next batch, the granules are thus obtained in a pretty uniform condition.

Scaling is a mode of treatment which is conveniently adopted for colloid salts; but it must be remembered that obtaining a salt in a nicely-scaled condition is no criterion of its quality. The percentage of the constituents may vary considerably without any conspicuous change in the appearance of the product, or amorphous foreign matter, such as gum, may be present without altering the physical character of the compound. The salts of peroxide of iron, with organic acids and the sulphate of beberine, are the principal medicinal substances kept in scales, but it has also been suggested to have extract of rhubarb and some other substances prepared in this form.

*Ferri et Ammoniae Citras.*

Peroxide of iron, freshly precipitated and washed, but not dried, dissolves pretty freely in solution of citric acid, but if the salt thus obtained is reduced to dryness, it is not again freely soluble in water. If, however, ammonia be added to the solution of oxide in citric acid, before it is evaporated, a rather indefinite compound of the two citrates is formed, which may be reduced to dryness without losing its solubility. Such is the nature of the well-known salt of the Pharmacopœia. If the oxide of iron be not in excess of the quantity prescribed, it will not be precipitated by either acids or carbonates of alkalies; but if it be much in excess of the quantity ordered, which may happen without changing the appearances, the addition of an acid will cause the separation of the iron, as a basic citrate, while the ammonia forms a salt with the acid added. The Pharmacopœia directs that the salt shall not leave less than 27 per cent. of peroxide of iron when calcined, and that the oxide thus obtained shall not be contaminated with alkali, which would result from potass or soda having been used instead of ammonia in the formation of the salt. It also directs that the citrate, when heated with excess of caustic potass, evolves ammonia and deposits iron, and the clear alkaline liquor from which the iron has been separated will not deposit cream of tartar if supersaturated with acetic acid, showing that tartaric acid has not been substituted for citric acid in its manufacture.

*Ferrum Tartaratum*

Is obtained by digesting fresh, moist peroxide of iron with cream of tartar, at a temperature not exceeding 140° Fahr., till no more peroxide of iron is dissolved.

The general characters of the salt thus obtained are similar to those of the citrate of iron and ammonia, but of course no ammonia is evolved when it is boiled with caustic soda, and if the alkaline liquor thus obtained be filtered from the per-

oxide of iron, and supersaturated with acetic acid, the cream of tartar originally used in the preparation of the salt is reprecipitated.

*Ferri et Quinæ Citras.*

In the preparation of this salt the peroxide of iron is dissolved in citric acid, the same as for making citrate of iron and ammonia; but before adding the ammonia some freshly-precipitated quinine—not the sulphate—is added, which forms citrate of quinine, and dissolves; but this would not form a permanently soluble salt without ammonia; consequently ammonia is added in small successive portions, each addition causing the precipitation of some quinine, which, however, speedily redissolves, unless too much ammonia has been added.

In the tests for this salt you will find the statement that it is precipitated white by liquor of ammonia, and brown by caustic soda; the latter, however, causes the separation of both white and brown precipitate—that is, quinine and peroxide of iron—while the ammonia precipitates the quinine white, and converts the yellow liquor into brown citrate of iron and ammonia.

It should also be noted that the commercial salt is frequently prepared with a deficiency of quinine, and sometimes with amorphous quinine, which will impart the full degree of bitterness, but will not yield the quantity of precipitate with ammonia which the Pharmacopœia directs.

All these three salts, when formed in solution, are to be evaporated at a gentle heat (not exceeding 140° Fahr.) to a syrupy consistence, and then spread upon plates of glass, and the desiccation continued till the salt is brittle enough to be detached in scales, which it usually does spontaneously.

EXAMINATION QUESTIONS.

What precaution is requisite in preparing solution of acetate of lead for crystallising, and what other salts require similar care?



If impure metallic bismuth be used for preparing the sub-nitrate, how are the different impurities affected by the process?

How is iron to be separated from chloride of calcium or chloride of zinc, if present as an impurity?

How is granulated sulphate of iron prepared, and what are its advantages?

What class of substances is it customary to have sealed, and what are the advantages of the process?

#### RECAPITULATION.

Some salts having a volatile acid or base, are liable to lose a portion of the volatile constituent during evaporation, and should be rendered neutral again before being crystallised.

Impure metallic bismuth dissolved in nitric acid, and poured into water, is precipitated; most impurities which might be present remain in solution; arsenic would go down with the bismuth.

Ferrous chloride is not readily separated from chlorides of calcium or zinc until converted into ferric chloride.

Granulated sulphate of iron is more easily obtained free from persulphate than the same salt crystallised in the ordinary way, and is less liable to become oxidised by keeping.

Scaling imparts a convenient form to colloid salts, but does not ensure their purity; it is therefore necessary to test their purity, especially in the case of citrate of iron and quinine, which is often deficient in quinine.

## LECTURE XIX.

### COMPLEX PROCESSES.

Some of the more Complex Processes of the Pharmacopœia—Tartaric Acid—Tannic Acid—Sulphate of Quinine—Hydrochlorate of Morphia—Resins of Jalap—Scammony and Podophyllum.

THE subjects which we have to consider at present belong more especially to the manufacturer; they are too complicated to be advantageously conducted upon the small scale, but they are nevertheless processes of high-class pharmacy, and though it is not expected of any retail pharmacist that he will conduct them for himself, no one can claim a good position for his profession who is not familiar with the principles upon which they are founded, and has some idea of the details and mode of operating.

As it will be impossible to go thoroughly into a number of complicated processes, we will take one or two for illustration, and only pass a few comments upon some of the others.

#### *Acidum Tartaricum.*

Cream of tartar, when boiled with water, and chalk added so long as effervescence takes place, becomes converted into tartarate of lime and tartarate of potass, the former being insoluble, the latter soluble. Chloride of calcium is then added, with which the tartarate of potass undergoes double decomposition, a further portion of tartarate of lime being deposited, while chloride of potassium remains in solution. The precipitate being washed till it no longer tastes of the chloride, is next to be decomposed with sulphuric acid, added in sufficient quantity to decompose the tartarate of lime, and the mixture

boiled for half an hour, or till it is judged that the reaction is complete. The deposit then consists of sulphate of lime, which is only sparingly soluble, and the solution contains the tartaric acid, with a small quantity also of sulphate of lime. As the solution is evaporated the lime salt is deposited, and separated by filtration before the liquor is finally reduced to the crystallising point. A further evaporation, till a pellicle forms upon the surface, prepares it for crystallisation. This crop of crystals usually contains still a small quantity of sulphate of lime, and is purified from this by again dissolving in water, filtering, and recrystallising. The mother liquors may be made to yield a second crop of inferior acid by further evaporation and crystallisation. The mother liquors are usually dark-coloured, and are sometimes bleached by the addition of nitric acid during the evaporation, by which the colouring matters are oxidised ( $\text{CO}_2$  and  $\text{NO}_2$  being evolved); but this endangers the contamination of the tartaric acid with oxalic.

The Pharmacopœia tests are intended to guard against the presence of oxalic acid, which would give a precipitate with sulphate of lime; and against sulphate of lime, which would give a precipitate with oxalate of ammonia; for though we need not fear the presence of *both* in one sample, we must guard against both separately.

The process for citric acid is very similar both in the general principles and the precautions required.

#### *Acidum Tannicum.*

Nutgalls contain 25 to 50 per cent. of tannic acid, which is freely soluble in water, spirit, or ether; but as the acid is very prone to decomposition, either by exposure to air or heat when dissolved in water, it is not practicable to extract it commercially by means of water. Ether is the solvent always used, and generally in combination with a small quantity of water. The process is one of simple extraction and evaporation, without any means of purification, the amount of impurity extracted by the ether being so small as to be of no importance

medicinally. A little colour and resinous matter are the only usual contaminations; the latter renders its solution in water turbid, but is of too small amount to interfere with the medicinal value of the product. As the aqueous solution undergoes spontaneous decomposition, tannic acid is kept dissolved in glycerine for medicinal use, or dissolved in spirit as a reagent.

### *Quinæ Sulphas.*

The British Pharmacopœia process for sulphate of quinine consists in macerating the *yellow* bark in dilute hydrochloric acid, by which the alkaloids are rendered soluble and are washed out by the subsequent process of percolation. The acid liquor being next precipitated with caustic soda added in excess, the precipitate which is deposited contains the alkaloids, together with a portion of the astringent acids of the bark (cincho-tannic, red cinchonic, kinic, and kinovic); which, however, are held more by mechanical attachment than chemical union, and are subsequently washed away by percolating cold water through the precipitate so long as it removes colour. As yellow bark contains very little of the other cinchona alkaloids (cinchonine and quinidine), the precipitate after washing consists of quinine, with only a small proportion of impurities. Caustic soda is preferred as a precipitant, because, when used in excess, it much promotes the washing out of the colouring matters, without the loss of quinine, which would be entailed by the use of caustic potass or ammonia.

Diluted sulphuric acid is then added, in a quantity just sufficient, or barely sufficient, to dissolve the quinine, with the aid of heat and a considerable bulk of water. The solution is filtered to separate a small quantity of resinous matter, which is left insoluble, and the filtrate evaporated, at or about boiling-point, till a film forms on the surface; it is then allowed to cool, when a considerable deposit of sulphate of quinine takes place. The sulphate of quinine is soluble in 30 times its weight of boiling water, but requires 750 times its weight of cold. There should, therefore, be 96 per cent. of the sulphate

of quinine deposited, and only about 4 per cent. retained in the mother liquor, along with the sulphates of the other alkaloids, should they be present; sulphate of quinidine being about 3 times as soluble, and sulphate of cinchonine about 10 times as soluble, as sulphate of quinine. Sulphate of amorphous quinine is also present in the mother liquor. The precise nature of this amorphous quinine is a disputed point; and so is the question of its original existence in the bark, or its production from the decomposition of some of the alkaloids during the process of extraction.

The proportion of sulphate of quinine yielded by different kinds of bark varies exceedingly, and even in different samples of the same species, *i.e.*, *C. Calasaya*, the product is said by Wittstein to vary from one to three per cent. The Pharmacopœia requires that yellow bark should contain not less than two per cent of pure quinine; but a manufacturer would be quite at liberty to use any inferior quality of bark from which he might find it profitable to extract quinine, provided the sulphate was ultimately obtained of the requisite purity.

The subjoined table shows the proportion of quinine and cinchonine yielded by several kinds of cinchona:—

<i>Quinine and Cinchonine in 100 parts of</i>			
<i>Brown and Grey Barks.*</i>			
Hunanoco, . . .	Quin.	0·40	Cinch. 1·70
Loxa, . . .		0·35	0·34
Pseudo Loxa, . . .		0·35	0·66
Huamalies, . . .		0·30	0·80
Jaen Pallida, . . .		0·56	0·60
<i>Yellow or Orange.</i>			
Calasaya,			
<i>a</i> Plana, . . .		2·20	0·28
<i>b</i> Convoluta, . . .		1·10	0·42
Fibrosa, . . .		1·05	0·83
Dura, . . .		0·54	0·48
Pitaya, . . .		1·68	0·90
<i>Red.</i>			
Cinchona rubra, . . .		0·91	1·05

\* Watts's "Dictionary of Chemistry."

*Morphiæ Hydrochloras.*

Several different processes have been devised for the extraction of morphia from opium, and separating it from the other alkaloids; it would not be profitable to occupy time with a full description of more than one, we will therefore confine our attention to the process of the Pharmacopœia.

One pound of opium is macerated in successive portions of water till it is exhausted, or we might more correctly say, till exhausted of its morphia, for it still retains a considerable part of the narcotine; the liquors being mixed and evaporated to one pint, deposit resinous and colouring matter and a further portion of narcotine; these being separated by filtration, the liquor contains meconates of morphia and other alkaloids;  $\frac{3}{4}$  of an ounce of chloride of calcium dissolved in water is now added; double decomposition takes place, by which meconate of lime is precipitated and hydrochlorates of morphia and codeia are deposited as a crystalline precipitate; the whole is concentrated so far that on cooling it becomes almost solid from the bulk of crystals which deposit; the crystalline mass is folded in a cloth, and strongly pressed to remove the mother liquor, which contains much colouring matter and hydrochlorates of codeia, &c., together, of course, with a portion of the morphia salt.

The cake of crystals contains meconate of lime and hydrochlorate of morphia and codeia; it is triturated with boiling water to dissolve the morphia (and codeia); the meconate of lime, being insoluble, is separated by filtration. The liquor, together with the water which has been used for washing the meconate of lime, is then evaporated again till it solidifies on cooling, and the pressure in folds of calico repeated to remove a further portion of impurities. If the morphia salt is still highly coloured, crystallisation is to be repeated again in the same manner. When sufficiently free from colour, the pressed cake is dissolved in six ounces of boiling water, and digested with quarter ounce of pure animal charcoal for twenty minutes, by which the remaining colour is removed. The pure morphia

is then precipitated by the addition of ammonia, the codeia remaining in solution. If the ammonia is added while the liquor is hot, the morphia is deposited in crystals as it cools, and is more readily washed clean; when the washings no longer give indications of chlorides with nitrate of silver, the morphia may be transferred to a dish or flask with two ounces of water, and, when it is brought to the boiling point, dilute hydrochloric acid added by slow degrees, so that the morphia may be dissolved with the smallest possible quantity of acid, that a neutral salt may result. As this solution now contains pure hydrochlorate of morphia, it may either be evaporated to dryness or concentrated and crystallised.

The following analysis of opium will give a general idea of the proportion of various constituents besides morphia, and shows how necessary it must be to have a well devised scheme to obtain a pure alkaloid from so complex a drug.

*Mulder's Analysis of Smyrna Opium.*

	Good.	Inferior.
Morphia, . . .	10·84	6·30
Narcotin, . . .	6·80	7·31
Codeia, . . .	0·67	·72
Narceine, . . .	6·62	9·00
Meconin, . . .	0·80	·58
Meconic acid, . . .	5·12	6·12
Fat, . . .	2·16	
Caoutchouc, . . .	6·12	
Resin, . . .	3·58	
Gummy extractive, . . .	25·20	
Gum, . . .	1·04	
Mucus, . . .	19·08	
Water, . . .	9·84	
Loss, . . .	2·14	

The pharmaceutical resins which do not belong purely to the department of materia medica are resins of jalap, podophyllum, and scammony.

*The Resin of Jalap,*

Or, as it is popularly called, jalapine, differs from the jalap resin of the old Edinburgh Pharmacopœia in being



more free from impurities. The process now includes washing with water, which removes small quantities of matter soluble both in spirit and in water. The old Edinburgh formula produced a true alcoholic extract, as the root was percolated with rectified spirit till all active matter was removed, and then the spirit removed by distillation, and ultimately evaporation in a water bath. But now,—the first part of the process remaining the same—water is added to the tincture, which causes the precipitation of the resin and facilitates the removal of the spirit, which thus distils off freely, the whole being recovered in the still. The water, at the same time, retains in solution some colouring matter and sugar. A further washing with two or three successive portions of hot water is enjoined to remove the last traces of sugar, which, from its being uncrystallisable and deliquescent, would otherwise impart an unpleasant stickiness to the ultimate product. The colouring matter is not thus completely removed; and if it is desired to obtain a pale-coloured resin, the powdered jalap may be mixed with animal charcoal in the percolator, or the tincture macerated with the charcoal, as a second stage in the process.

#### *Resin of Scammony*

Is obtained in precisely the same way from scammony root or from the inferior qualities of commercial scammony, by which means they are freed from any earthy or amylaceous impurities they may contain.

The commercial resin of scammony, prepared from the root, is usually contaminated with a portion of deliquescent matter, present from imperfect washing of the resin. It is detected by macerating the sample in ether, which dissolves the resin, but leaves the watery extraction. If this impurity is present in any notable quantity, it imparts a stickiness to the resin which pure samples do not possess. If there be but a small quantity of the aqueous extractive present, it is only detected by its insolubility in ether.

*Resin of Podophyllum.*

Commonly known as podophyllin, is obtained by a similar process, but the distillation of greater part of the spirit is directed without the previous addition of water.

If the concentrated tincture, as it comes from the boiler of the still, be added to water, the resin is precipitated, leaving berberin in solution; but as hydrochlorate of berberin is insoluble in water, the Pharmacopœia directs that the water used for the precipitation of the resin should be mixed with one twenty-fourth of its bulk of hydrochloric acid, that the hydrochlorate of berberin may be precipitated along with the resin. It is said that much of the activity of podophyllin depends upon the presence of berberin; it is, therefore, important to ensure the presence of this active principle by the addition of the acid to the water.

It is also important to examine imported samples, as most of them come from America, where, according to Professor Parish's statement in 1864, the practice of manufactures was not uniform in this respect, the U.S. Pharmacopœia not directing the use of acid, and the test of purity indicating the absence of the berberin, thus—"It is partially soluble in ether, and the residue, when dissolved in solution of potassa, is not precipitable by hydrochloric acid."

The British Pharmacopœia tests require that it should be soluble in rectified spirit and in aqueous solution of ammonia, that it should be precipitable from the alcoholic solution by water, and from the alkaline solution by acids; these reactions take place both with the resin and the hydrochlorate of berberin.

If the podophyllin be boiled in dilute acetic acid, the resin remains undissolved while the berberin is taken up, and, as liquor cools, it is redeposited in the form of a yellow precipitate.

Contrary to Parish's requirements, our podophyllin, when exhausted with ether, leaves a residue soluble in solution of potassa, and which *does* yield a precipitate on being neutralised with hydrochloric acid.

## EXAMINATION QUESTIONS.

Describe the leading features of the process for tartaric acid, and the impurities to be guarded against.

What are the principal impurities to which tannic acid is subject?

Describe the leading features of the process for sulphate of quinine, and the mode of removing the colouring matter.

Describe the morphia process, giving the objects attained by the different stages.

What is the general process for pharmaceutical resins, and in what respect does the process for podophyllin differ from those for resins of jalap and scammony?

## RECAPITULATION.

Tartaric acid is prepared by converting cream of tartar into tartarate of lime, and then decomposing the tartarate of lime with sulphuric acid. The tartaric acid is liable to be contaminated with sulphate of lime, or, if nitric acid has been used to oxidise the colour, it may be contaminated with oxalic acid.

Tannic acid is obtained by extracting galls with ether, and a little water; the principal impurities are colouring matter and a little resin.

Sulphate of quinine is obtained from yellow bark by extraction with water and  $\text{HCl}$ , precipitating with  $\text{NaO}$ , and dissolving the washed precipitate in hot water with the least possible quantity of sulphuric acid.

Hydrochlorate of morphia is obtained by decomposing infusion of opium with  $\text{CaCl}$ , evaporating the liquor to small bulk, pressing the resulting crystals, repeating the crystallisation and pressing, dissolving the crystals, precipitating with ammonia, and dissolving the washed precipitate in water and  $\text{HCl}$ .

Resin of jalap is precipitated from a tincture of the root,

he spirit being distilled off, and the residue washed with water.

Resin of scammony is produced the same way.

Resin of podophyllum is precipitated from the tincture by the addition of water and HCl; the latter precipitating the berberin, which would be washed away if this addition were not made.

## LECTURE XX.

### DISPENSING.

Importance of Orderly Habits—Preparations in Anticipation of Requirements—Prescriptions difficult to read—Weighing—Measuring—Ambiguous Prescriptions, Unusual Doses, &c.—Modes of Mixing—Soluble Salts—Precipitation of Active Matter—Use of Distilled Water—Modes of Mixing Powders in Suspension—Filtration—Spirit of Nitrous Ether to be Neutralised—Emulsions, Egg Liniments, &c.

PRACTICAL dispensing requires for its satisfactory and successful prosecution a considerable versatility of talent. The dispenser ought to have a good fund of general information—information, it may be, which is only rarely called into requisition, yet may be essential to success on some particular occasion. Every day may call into play some knowledge which will not be wanted again for months; there are constantly opportunities arising for making observations which will be useful again at a future time, but it may be months, or years, or only hours, before the opportunity for their use may recur. It is this uncertainty which makes the task of observing, and profiting by our observations, a tedious and difficult one, and makes dispensing an art which can only be perfected by long experience, and a constant disposition to profit thereby. Something may be learned by books, something by verbal instruction, and something by practical demonstration; but these are trifles compared to what must be learned by personal experience and observation.

What information I have to impart will be most valuable to you as illustrating what you may acquire for yourselves.

There is no department of business in which the formation of good habits is so important as in dispensing. To do a thing by habit requires no attention, and gives no trouble after the habit is once formed. If you have the habit of putting your measures, mortars, knives, &c., away dirty, or leaving them about, because they are not clean, there will be every probability that they will give twice as much trouble to clean when wanted, and will also risk the spoiling of some medicine by being used without cleaning. If you form the habit of putting things down just where you have used them, or just where it is most convenient at the time, it will probably cost you ten times as much trouble to find them next time they are wanted; whereas, once form the habit of putting them in their right places, and doing so no longer costs either thought or trouble. You perhaps think that I am only telling you what every one knows. Be it so; I may add, I am only telling you what very few put in practice. It is easier to find a classical scholar, an accomplished chemist, or a learned botanist, than it is to find a man who both knows and practises those moral qualifications to which I have been alluding; and, while this is the case, I feel that I cannot omit the inculcation of those habits which add so much to the value of a dispenser, and which, as far as my own experience goes, are so rarely to be met with.

The most commonplace man can have his house in order when he expects any one coming to inspect it, or will perform his work well on any great occasion; but the rare and valuable qualification is the habit of doing the same on small occasions, and on *all* occasions. Let it be your habit—a habit so strongly formed as to be your second nature—to be orderly, accurate, neat, and quick, attentive to little things, observant of everything. There is commonly more time wasted in going through the work in a spiritless manner than would suffice to acquire the theoretical knowledge in conjunction with the practical experience which we too often find wanting, and the absence of which is excused on the plea that no time has been

allowed for its acquirement. The time spent in putting things in order, seeing that everything is clean and in its place, and ready for the next job, is about the best spent portion of the day. Nothing can be more idle or pernicious than the idea that it is beneath the dignity of a dispenser to do this kind of work for himself—it is only the sham gentleman who is afraid of losing caste by attending to those little things which really add so much to his own value. He who keeps his own department in nice order will soon learn to do his work with the least possible dirt, either to his appliances or to himself, and will add to his accuracy at the same time. The dispenser should be an accomplished man and a gentleman,—up to every duty, and above none.

I might enlarge upon many points, but I hope I have said enough to be a useful hint to those who are willing to take it. I must now pass on to a few aids to dispensing.

There are many operations which may be conveniently performed in anticipation of their requirement, or may be facilitated, by some preparation during leisure moments to save those more precious moments when business is pressing.

Salts are usually kept in powder, which facilitates their solution. Some may be conveniently kept ready dissolved, such as sulphate of magnesia; 10 ounces being put into a pint bottle and filled up with warm water, will make a convenient solution, which does not crystallise at ordinary temperatures, and of which a fluid ounce may be measured, instead of weighing half an ounce of the crystal and having to dissolve it at the time of dispensing.

Bicarbonate of soda and potass may be dissolved in a similar way, the solutions most conveniently containing 4 gr. of the former or 10 of the latter in a fluid drachm. In all these cases the contents of the fluid drachm or ounce should be conspicuously marked upon the label.

Calcined magnesia is sometimes mixed with water in the proportion of 3j. in fl.ʒj., but if not rapidly used it is apt to



agglomerate, forming lumps so hard as to give trouble instead of saving it.

Iron mixture may be conveniently kept ready made, all but the addition of the sulphate of iron, and should be labelled in this style—

The addition of  $2\frac{1}{2}$  gr. sulphate of iron to  
1 ounce of this mixture, makes  
MIST. FERRI CO. B.P.

The dry ingredients of chalk mixture may be kept mixed, and labelled—

One ounce of this powder with  
 $7\frac{1}{2}$  ounces of cinnamon water, makes  
MIST. CRLTÆ CO. B.P.

The formulas for various preparations not very often wanted, or which do not keep well enough to be worth making in anticipation of the requirement, may be kept written out for different quantities, to save calculation or the chances of error, by having the same to do in the hurry of business. Thus—

*Mist. Guaiaci.*

R. P. Resin Guaiaci,	.	.	$\frac{1}{2}$ oz.	88 gr.	11 gr.
Sacchari,	.	.	$\frac{1}{2}$ oz.	88 gr.	11 gr.
P. G. acaciæ,	.	.	$\frac{1}{4}$ oz.	44 gr.	$5\frac{1}{2}$ gr.
Aq. cinnamomi,	.	.	20 oz.	8 oz.	1 oz.

*Confectio Piperis.*

R. P. Piper nigr.,	.	.	2 oz.		44 gr.
P. carui,	.	.	3 oz.		66 gr.
Mel. despumat,	.	.	15 oz.		$\frac{3}{4}$ oz.
			20 oz.		1 oz.

The selection of the formulas which it is desirable thus to divide depending, of course, upon the demand there may be for one or other of the preparations.

It is also convenient to have some extracts, such as colocyath, aloes, and rhubarb; and some pill masses, such as rhubarb, assafœtida, and compound calomel, in powder as well as massive. The extracts, however, it must be remembered, are in some cases injured by drying, becoming less soluble, and probably less active.

The most important of all these aids to despatch are the concentrated infusions and decoctions. Unfortunately, they are not sanctioned by the authorities; and whatever may be their medicinal value, they are not readily made to have precisely the same physical qualities as the fresh; but while in theory almost every one prefers the fresh infusions, in practice almost every one will more or less frequently use the concentrated. I will not attempt to teach you to make them, but must remark, in passing, that none should be used which do not afford a pretty close representation of the fresh as determined by a comparison with a sample of fresh made for the purpose.

We may now suppose your dispensary is in order, and a customer presents a prescription. Your first duty is to read it, which must be done accurately, yet so promptly as not to leave any impression upon the mind of the patient, either that it has been carelessly written or that you are dull in comprehension. In most cases there will not be any difficulty, but occasionally there will be some little hitch, which is detected by glancing over the paper, while you ask the patient if you should send the medicine, or if he will call back for it; and if perchance he prefers to wait for it, you will have to make up your mind very promptly what is to be done under the circumstances, and this must depend very much upon the nature of the difficulty. If it is simply a case of bad writing, you will probably overcome it without alarming the patient, by commencing to copy the prescription and write the label; the time gained in doing this, and the careful examination of each word, and comparison of doubtful words with others, will generally clear up the question. Never hesitate to ask

the aid of some one else when it can be done, even if it be only to confirm your own reading; and when you have hit upon a probable interpretation, see that it agrees in all respects with the other part of the prescription—*first as regards the style of the letters, then as regards the nature of the medicine, and finally as regards the suitability of the dose.* If, after all consideration, a safe and satisfactory conclusion cannot be arrived at, an appeal to the prescriber may be necessary, and care should be taken not to give any avoidable alarm to the patient. It is best to see the doctor without the patient knowing it. If the doctor is not within reach, a candid, open confession of the difficulty is usually the best course. Sometimes the patient himself can aid, if he has had the medicine before, and can say what it was like, or what it is for, &c. In all cases of doubt or difficulty, when overcome, a note should be made to avoid the same doubt occurring a second time. Some pharmacists make a practice of keeping doubtful prescriptions, and giving the patient a copy; others make a marginal note to indicate their reading; a third class will copy the prescription, making a note in the prescription book. Whether the first and second be approved or not, the last should never be neglected. Whether the difficulty be in reading or dispensing, the prescription book should contain such notes as would prevent any chance of making a repetition different from the first.

*Labelling* should be done in a neat, plain, legible style, without any attempt at ornament or flourish, and should state briefly and clearly what is necessary, with the native idiom and conventional expressions.

*Weighing.*—The dry materials of a mixture should be carefully weighed. Such a thing as guessing an ingredient should be so far from your thoughts as not even to require the thought that you will not do it. The scales should be kept in good condition, the pans always clean, and wiped out after each time of using. Any material apt to act upon the pan chemically, or to adhere to it, should be weighed upon paper. For

this purpose take a piece of smooth paper, double it, and while double cut it with scissors into two circular pieces, which will now be placed one on each scale pan, and will no doubt balance one another. The weight being placed on one paper, and the material being weighed on the other, the weighing will be effected accurately without the material touching the pan. This mode should be adopted for soft extracts, deliquescent or corrosive salts, and for iodine. Much trouble will be saved in the end by the use of this simple precaution.

*Measuring.*—The fluid ingredients of a prescription are always estimated by measure, unless specially directed to the contrary. The ounce or drachm mark without the “fl.” before it, literally means an ounce or drachm by weight; but if an ounce each of ether and chloroform were prescribed, thus—

R	Æther,	.	.	.	.	.	℥i.
	Chloroform.,	.	.	.	.	.	℥i.
M.							

a fluid ounce would be understood, though the fluid ounce of ether weighs  $\frac{3}{4}$ ths of an ounce, and the fluid ounce of chloroform weighs  $1\frac{1}{2}$  ounce.

Graduated measures usually have, and ought always to have, graduations on both sides; and in using the measure it should be held a little below, but almost on a level with the eye, so that it may be seen that the edge of the liquid meets the graduation at the back of the glass at the same moment as that at the front. If this be not attended to the glass may not be level, and consequently more or less than the legitimate quantity may be contained in it, while the graduation on the front appears to indicate the quantity required.

When a number of fluids have to be measured into a mixture, the dispensing bottle should be carried in the right hand to the different parts of the establishment where these fluids are kept, the measure glass being held between the thumb and the fore finger and second finger of the left hand, and the prescription between the two middle fingers of the left hand.

The dispensing bottle may then be set down beside the stock bottle of the tincture or infusion required, while the stock bottle is lifted by the right hand, and its stopper removed by the little finger of the left hand. After the quantity has been measured, the stopper may be replaced, the bottle returned to the shelf, and the dispensing bottle being again lifted by the right hand, the contents of the measure transferred to it. In this way a succession of quantities may be accurately measured with the least possible consumption of time, with the prescription always under the dispenser's eye, and without any stock bottle being carried away from the part of the shop which it usually occupies.

By attention to such little points as these, accuracy, order, and quickness may be much promoted.

We will now turn our attention to a variety of cases which, occurring to me in actual practice, have presented some little difficulty, and which will sufficiently serve to illustrate what is likely to be met with by any one who has much dispensing to do.\*

R	Mist. amygdal.,	.	.	.	gtt. xvi.
	Acid. hydrocyan.,	.	.	.	℥viii.
M.	Capt. cochl. ij.	bis vel ter die.			

This is an example of misplaced quantities, and though it is a mistake of the utmost gravity, it is so evidently a case of simple misplacement that no hesitation could be felt about making it up with the quantities reversed.

R	Potass. iodid.,	.	.	.	℥ss.
	Aquæ camph. ad.	.	.	.	℥vi.
M.	Sumat. ℥ss.	3tiis horis.			

28,238.

The British Pharmacopœia dose of iodide of potassium is five to ten grains; in this recipe we have twenty grains every three hours. But we are not bound on that account either to

\* The correspondence column of the Pharm. Journal will frequently supply further illustrations of difficult cases, and how the difficulties are to be met.

suppose that half a drachm had been intended, or to see the physician and make sure of the half ounce being his intention. Such a dose is not officially recognised, and not commonly prescribed; but we must also add, is not dangerous, and is occasionally given; it may therefore be dispensed as written, without question.

R	Sp. æth. nitros.	.	.	.	.	℥iiss.
	Chlorodyn (C.B.),	.	.	.	.	gtt. x.
	Vin ipecac.,	.	.	.	.	M. xx.
	Aquæ camph. ad.,	.	.	.	.	℥ii.
M.	Ft. Haust hora somni sum.					
	31,886.					

In this draught the dose of spirit of nitre is larger than usual, the British Pharmacopœia dose, being ℥ss to ℥ij; but that need give no uneasiness, as the excess prescribed is not large, and the preparation is not a dangerous one.

R	Magnes. carbon.,	.	.	.	.	℥iii.
	Rhei pulv.,	.	.	.	.	℥i.
	Zingib.,	.	.	.	.	℥i.
	Tinct. card. co.	.	.	.	.	℥i.
M.	Capt. cochl. iij. omni mane.					

This is an evident case of omission of the excipient; but there being no clue to the nature of the material which was to give bulk to the mixture, and no clue to the bulk intended, there was no course open but to see the prescriber and ascertain that "Aquæ ℥vij." had been omitted.

R	Potass. iodid.,	.	.	.	.	℥i.
	Magnes. sulph.,	.	.	.	.	℥vi.
	Inf. quassiae. ad.,	.	.	.	.	℥viii.
	Syr. aurant.,	.	.	.	.	℥ss.
	Aquæ ad.,	.	.	.	.	℥viii.
	Sumat. ℥ss. ter die.					

28,250.

This is an equally clear case of adding water when it was unnecessary, and as it was evidently the intention to make an 8-ounce mixture, the infusion of quassia was added in sufficient

quantity for this purpose, and the water simply omitted without troubling the doctor or patient about the matter.

R	Mur. hydrarg.,	.	.	.	.	gr. ss.
	Mur. ammoniæ,	.	.	.	.	gr. v.
	Extr. belladonnæ,	.	.	.	.	gr. viii.
	Alcohol,	.	.	.	.	ʒi.

Tere simul et adde aqua bullient ʒviii., et cola per chartam.

20,007.

As a lotion to the eyes.

In this prescription the question arises what is "Mur. hydrarg.?" Though in most cases it would be understood as calomel, there is here no question that corrosive sublimate was intended, the "Mur. ammon." being added to promote its solution. Calomel would have remained undissolved, and consequently separated by the filtration.

R	Hydr. chlorid.,	.	.	.	.	gr. ii.
	Extr. colocynth co.,	.	.	.	.	gr. viii.

Misce, fiat pilula sumut duo pro re nata.

Mitte xxxvi.

20,149.

The quantity ordered here to make a pill being unusually large, it was a question whether it had not been the intention of the prescriber to insert a number—say 2 or 3 after the word pilula. The patient being a lady not in the habit of taking very strong medicine, this was decided upon, and it was made into two pills as the most likely.

R	Tinct. mel. vesicatoriæ,	.	.	.	.	{ āā
	Liq. potass.,	.	.	.	.	{ ʒss.
	Tinct. kino,	.	.	.	.	ʒv.
	Bals. copaib.,	.	.	.	.	ʒi.

M. A teaspoonful in water three times a-day.

28,627.

Mel. is usually translated honey, but as this did not make sense of it, an appeal to the *Materia Medica* cleared up all doubt, by showing that *Melœ vesicatoria* was the name by which Linnæus designated *cantharidæ*s.

Contractions are a fruitful source of doubt. I have met with



various instances, such as "Acid. hydroc." which might be hydrochloric or hydrocyanic. If it were in company with nitric acid and taraxacum juice, we might conclude that hydrochl. was intended. If it were given in almond emulsion, or other remedies for cough, hydrocyanic would be understood; always, however, having regard to the propriety of the dose. "Inf. sen." may be infusion of senna or of senega, and to determine it we will be guided by the probability of a purgative or an expectorant being intended. "Potass. sulph." may be sulphate or sulphuret. If a few grains are ordered internally, sulphate would be our reading. If four ounces were ordered for a bath, sulphuret would no doubt be the intention. "Sodæ carb." is generally understood to be "Bicarb.," but I once met with a case where "Sodæ carb. pulv. ʒij." was ordered for a bath, and as the word "Pulv." was doubly underlined, it was supposed to indicate that the carbonate was to be used which is not usually kept in powder, *i.e.*, the monocarbonate, not the bicarbonate.

Instead of further multiplying examples, I will just add a caution, never to overlook any circumstantial evidence which will throw light upon instructions which in themselves are not sufficiently explicit.

After the task of interpreting the prescriptions comes the problem of how to mix the medicine in a suitable condition. Ordinary mixtures rarely present any difficulty; but, as exceptions will be met with occasionally, I will adduce a few illustrations, together with a few general hints on convenient methods of mixing.

Our aim must be to make the mixtures uniform, that is, the same mixture should always have the same effect, and look, taste, and smell the same, and there should be little or no difference between the first dose the patient takes and the last.

Soluble substances should always be dissolved before the medicine is put into the patient's hands, *except* when there is more than can be retained in solution at ordinary temperatures. The use of warm water is often a great convenience in expediting the solution of such salts as chlorate of potass, borax, or

alum, which only dissolve slowly ; but if the mixture can only retain a portion of the salt in solution, when it is again cold the remainder will be deposited as crystals on the sides of the bottle, in a condition in which it will be almost impossible for the patient to take it. If, therefore, the salt be in excess of the solvent, it must be reduced to the finest possible state of division, and the direction to shake the bottle must be added.\*

I have here two mixtures containing one ounce bicarbonate of soda in eight ounces of water, one of which was made with hot water and complete solution effected; as it cooled, however, a portion of the salt was deposited in crystals on the side of the bottle in a most inconvenient manner; the other was rubbed as fine as possible with cold water, and was while new (two years ago) as good a mixture as could be made of the materials. I showed it to you last year, and it had then become knotted and hard with twelve months' keeping. You will observe, now that it is two years old, it has become distinctly crystalline.

It would be well for medical men to make a rule as far as practicable to give soluble substances in a sufficient quantity of solvent to retain them perfectly in solution at ordinary temperatures ; but as we cannot regulate this point, our next duty is to have the mixture in the most permanent condition we can give it when it leaves our hands, *i.e.*, as much and no more dissolved than it can retain under ordinary keeping.

Next to these we may consider cases in which one chemical may precipitate another.

	31,768.	
R Potass bicarb.,	.	℥iii.
Liq. strychniæ,	.	℥ss.
Aquæ ad.,	.	℥viii.
M. Capt. ℥ss. ter die ex aqua.		

Strychnine acquires 6667 parts of water for solution at ordinary temperatures; as here is quarter of a grain in 3500 grains of water, we need not fear its precipitation.

\* See solubility of salts in Storer's " Dictionary," &c.

31,734.

R	Morphiæ hydrochl.,	.	.	.	gr. i.
	Acid acet. dil.,	.	.	.	℥ xv.
	Liq. ammon. acet.,	.	.	.	℥i.
	Aquæ ad.,	.	.	.	℥ii.
M.	Ft. Haust.umat, ℥i. omni nocte.				

The addition of acetic acid in this case is no doubt intended to insure the freedom from carbonate of ammonia which might be present in the acetate of ammonia if not accurately neutralised, and which would precipitate the morphia. If the addition of 15 drops of diluted acetic acid failed to make the liquor distinctly acid, it would be legitimate to add more, following the spirit rather than the letter of the instruction.

R	Sodæ boratis,	.	.	.	℥ss.
	Morphiæ hydrochlor.,	.	.	.	gr. vi.
	Aquæ,	.	.	.	℥viii.
M.	Ft. lotio.				

The two salts being dissolved separately and mixed, the morphia was precipitated. Being an external remedy it was not unsafe, and was supplied to the patient in that condition and a note was sent to the prescriber informing him of the decomposition, that he might, if necessary, modify this combination in subsequent prescriptions.

Distilled water should be used as a regular habit; there are many cases in which it makes no important difference in the medicinal value of the mixture, but on which it still makes a difference in the appearance, and we must remember that the appearance will sometimes have a great influence upon the patient, and it is well, when our work is called in question, to be able to show that our practices will bear the strictest investigation. Infusions and sundry other clear preparations made with distilled water will give no precipitate with alkaline carbonates; but if hard water were used, carbonate of lime would be deposited, and the patient getting his mixture clear at one place and turbid at another, would naturally suspect

that the latter was carelessly made or of inferior materials. I have here three mixtures illustrating this point.

R	Sodæ bicarb., . . . . .	℥ss.
	Aq., . . . . .	℥iv.
M.		

This being made with hard water, is turbid; had it been made with distilled water, it would have been bright. If the patient were to show me the prescription and ask the question, I could not deny that the preparation was at fault.

Take another :

R	Liq. arsenicalis, . . . . .	℥i.
	Liq. potassæ, . . . . .	℥ss.
	Aq. ad., . . . . .	℥iv.
M.		

In this case I have two mixtures, one made with "Aq. font." and the other with "Aq. dest." You observe the latter is quite bright, but the former contains a dirty pink sediment, which the patient will say is the arsenic not properly dissolved. You will say it is carbonate of lime previously held in solution in the water by excess of carbonic acid which the potass has abstracted; and the lime salt has gone down in combination with the colouring matter of the liquor arsenicalis. You must admit, at the same time, that it should not be there, and however well you may be satisfied that it is of no moment, the impression on the patient's mind will be anything but satisfactory. You may suppose that I am making more of this than there is good grounds for, but I experienced a case which will show you its importance. A prescription came to me containing, besides other matters, a small quantity of compound tincture of cardamoms and a bulk of water; as I sent the medicine out it had a pale red colour. The patient returned it to me, saying that there was some mistake, as it was not the same as he got from one of my neighbours. On inquiry, I learned that what he previously had was of the violet pink tint which the tincture of cardamoms makes with an alkaline solution. I explained to him that the prescription

contained no alkaline matter, and the colour might be changed in his previous supplies by the use of hard water. I mixed a little of the tincture in two glasses, one with hard and the other with distilled water, and showed a difference in tint, and thus established his confidence in my accuracy. But enough of this; use distilled water at all times.

Some vegetable powders, such as rhubarb and ginger, if simply put into the bottle with a watery excipient, are apt to remain for a long time in dry knots, to avoid which they may be rubbed in a mortar with a small portion of the liquid; or, having been rubbed smooth, without moisture, but advantageously with any saline ingredient, they will mix readily if simply put into the bottle half full of water or infusion and briskly shaken. The froth formed in this way is sometimes troublesome, especially with decoction of aloes or sarsaparilla. If a tincture is to be added, this will usually extinguish the froth; if no tincture is contained in the mixture, it may be poured through a funnel into another bottle, and the froth will remain behind; but it is better to avoid making the froth, which may be done by using so little of the fluid to shake the powder with that it makes a creamy consistence first, which may be afterwards diluted without agitation. Or the mixing may be effected in a mortar. Or, if a tincture be present, the powders may be diffused in it first without the liability of frothing, and the aqueous fluid added afterwards. Ginger and other oily and resinous powders mix with spirit much more readily than with water; but pure resins, soluble in spirit, are apt to run into adhesive lumps if put into spirit, which they are not liable to if put into water. Whatever the powder may be, it is best to put some fluid into the bottle first, that the dry materials may not stick to the bottom.

There are many mixtures which undergo some slight change, such as the deposit of a resinous matter from a tincture when it is added to an infusion; in such cases it is the custom of some establishments to filter the mixture before it is sent out, a practice, however, which I must strongly condemn, as valu-

able constituents may frequently be thus removed. This is the case when tincture of Indian hemp is added to water, or tincture of quinine to infusion of roses, also when spirit of juniper, or other solution of essential oil, is added to a watery fluid. It would be better if medical men would avoid ordering mixtures of that kind, but, when ordered, the pharmacist's duty is to make them up as directed, however unsightly they may appear, and not filter unless he is directed to do so.

R	Tinct. belladon.	.	.	.	.	ʒi.
	Syrup. ferri iodid.,	.	.	.	.	ʒi.
	Potass. bicarb.,	.	.	.	.	ʒi.
	Aqua camph. ad.,	.	.	.	.	ʒvi.
30,119						

The above mixture was probably prescribed unmindful of the decomposition which would occur, but if the pharmacist were to filter it, on the ground of its not being intended to contain a sediment, he would be taking an unwarrantable liberty with its therapeutic action.

When spirit of nitrous ether is ordered with iodide of potassium, a small but uncertain quantity of iodine is always liberated from the action of free acid contained in the spirit of nitre, to avoid which the nitre may be first neutralised with carbonate of potass. I have seen a mixture containing compound decoction of linseed, iodide of potassium, and spirit of nitre, turn purple-black from the iodide of starch formed in this way. The mixture being once black could not be rectified, but if the nitre was neutralised first, the formation of colour was quite prevented.

The production of emulsions is probably the task in which the pharmacist requires most skill and judgment.

An emulsion consists of oil or resin suspended in a watery fluid in such a manner as to have a milky appearance, and not readily to separate. The union of the oil with the water is effected by the intervention of alkali, soap, gum, emulsine, pancreatine, &c. Emulsion of almonds is so simply made as to

require no comment. Emulsions of the gum resins, such as myrrh and ammoniac, are also made by simple trituration of the dry material with water gradually added; attention should be had to the condition of the gum—fresh, soft, “cheesy” gums making the best emulsions. Powdered or dried gums should not be used.

Castor oil, almond oil, and balsam of copaiba are emulsified by the aid of gum, alkali, or soap, or the use of both gum and alkali.

The mixture is most perfectly effected when the oil and water are present in about equal bulks. The best mode of operating is to put the gum and water or mucilage into a mortar, and add the oil or balsam by degrees, with brisk stirring; if the mixture becomes thick with the addition of the oil, water should also be added before more oil, so as to maintain the emulsion during its formation at about the consistence of a thin cream. When the mixing has been thus effected, the remainder of the liquids are to be added, taking care that tinctures are added last, as they are liable to cause the separation of the emulsion into its constituent fluids.

An emulsion may be made with less trouble, though not quite so perfectly, by putting the oil or balsam into the bottle with powdered gum, shaking them together till mixed, which takes place very readily, then adding about double the bulk of water, and shaking briskly.

Alkaline emulsions are usually made without the mortar by putting into the bottle the alkali with a measure of water equal to the oil which is subsequently to be added, emulsification may then be effected by brisk agitation, the remaining fluid being gradually added.

Liquid extract of male fern is frequently prescribed with mucilage, with which it does not make a very satisfactory emulsion; a better form of administration is to emulsify the extract with a little alkali, or soap and mucilage, before adding the bulk of the vehicle. There appears to be some prejudice against the use of soap in making emulsions for internal use; it might, however, be much more frequently applied with ad-



vantage, and the olive oil soaps are now made so free from objectionable taste as to be quite suited for this purpose.

Mixtures analogous to emulsions frequently occur from the addition of resinous tinctures to water; the separation of the resin in a sticky condition, in which it may cohere into lumps or adhere to the bottle, will frequently be the result if gum be not added, while a permanent emulsion may be obtained by its use.

Take the following as examples:—

R	Ammon. hydrochlor.,	.	.	.	℥ii.
	Potass. chlorat.,	.	.	.	℥ii.
	Tr. laricis,	.	.	.	℥iii.
	Pulv. tragacanth,	.	.	.	℥i.
	Aqua, ad.,	.	.	.	℥viii.
M.					29645.

The resinous matter of the larch bark would be separated on dilution of the tincture, were it not for the presence of the tragacanth, which, though it does not prevent its precipitation, prevents its agglomeration or subsidence. On the other hand, we may notice that there is difficulty in making a smooth solution of tragacanth by adding the powder to water, either in the bottle or mortar, but it diffuses with perfect freedom in spirit; and the subsequent addition of water with agitation makes a smooth mucilage without difficulty; the best method of making the above mixture being to put the tincture into the bottle, add the gum and salts, shake together, add half the water, shake again, and then fill up with the remainder of the water.

The next prescription is an example of the difficulty arising from want of gum.

R	Mist. camph.,	.	.	.	℥vii.
	Tinct. camph. co.,	.	.	.	℥i.
	benzoin,	.	.	.	℥iii.
	scillæ,	.	.	.	℥ss.
M.					

The gum benzoin separated as a clotted mass, in a condition quite unfit for the patient to take; and this was the same

whatever mode of mixing was adopted, until the expedient was tried of adding 20 grains of powdered tragacanth to the mixed tinctures, and then adding the water with brisk agitation. A perfect emulsion was the result. On showing the two mixtures to the prescriber, he most willingly sanctioned the addition.

Though powdered gum may be added to alcohol without any fear of its becoming knotted, the case is somewhat different with mucilage; if the latter be poured into strong spirit, the gum separates in clotty adhesive flocculi, though the mucilage will bear a considerable addition of spirit, if the spirit be poured in gradually while the mucilage is kept stirred. We avail ourselves of this in making such mixtures as the following :—

R	Tinct. guaiac. am.,	.	.	.	}	āā
	Mucilag. acaciæ, .	.	.	.	}	īi.
	Potassii iodidi, .	.	.	.		ḡii.
	Tr. arnicæ, .	.	.	.		īiii.
	Decoct. cinchon. ad.,	.	.	.		īviii.

M.

The mucilage is to be put into the mortar, and the tincture added by degrees, with constant stirring. When about a third of the tincture has been thus added, the mixture is thicker than the original mucilage, and should have a small addition of the decoction of bark to thin it, and then more of the tincture. In this manner the whole of the guaiacum may be suspended without any of the acacia being precipitated; the other ingredients being added, a perfect mixture is formed.

White of egg and yolk of egg are both used for converting essential oils into emulsions with watery fluids for use as liniments. The following is a form we have many times made, and fairly represents the class :—

R	Ol. terebinth,	.	.	.	.	īss.
	Ol. origani.,	.	.	.	.	īiss.
	Vitelli ovi.,	.	.	.	.	q. s.
	Liq. ammon.,	.	.	.	.	īiii.
	Aceti, .	.	.	.	.	īiv.

Ft. liniment.

The yolk of egg is best rubbed in the mortar with the ammonia first, the alkali helping to dissolve down any stringy particles. When this is sufficiently accomplished the oils are to be added, with constant stirring; and should the mixture become thicker than cream before the whole of the oil is mixed in, a little of the vinegar should be added to thin it again before adding the remainder of the oil; and when the union is satisfactory, the rest of the vinegar mixed in by degrees. This makes a good emulsion, and keeps well if the acid be in excess, but is apt to become putrid with keeping if the ammonia preponderates.

Much more might be said upon this subject if time allowed; but the principles and mode of operating just described will be found to apply pretty readily to many modifications which will occur in practice.

#### EXAMINATION QUESTIONS.

When an ambiguous word or quantity occurs in a prescription, how would you determine which of several readings was the most probable?

If an ounce of chloroform be ordered in a prescription, should it be dispensed by weight or measure?

If you find a medicine ordered in doses larger than usual, what considerations would guide your proceeding?

What rule should be observed in dispensing soluble salts in mixtures?

What rule should be observed regarding the filtration of mixtures?

What advantages are there in the use of distilled water?

#### RECAPITULATION.

Habits of order and of observation are most important to the dispenser.

When an ambiguous word occurs in a prescription, and a probable reading has suggested itself, compare it with the other parts of the prescription as regards the form of the let-

ters, the nature of the medicine, and the suitability of the dose.

When difficulties are overcome, make such notes as will prevent their recurrence.

In dispensing, all fluids except mercury are estimated by measure, unless the contrary is indicated; as a rule, "solids by weight, liquids by measure."

No strict rule can be laid down regarding over-doses. Doses larger than usual may be dispensed as written if they do not involve danger to the patient; any dose apparently unsafe should be corrected or confirmed by the prescriber before being dispensed.

When a contracted word is capable of two interpretations, judge of the drug by its companions.

Soluble ingredients in a mixture should be entirely dissolved before the mixture is sent out, provided they will remain in solution under ordinary keeping.

Mixtures naturally turbid should not be filtered unless instructions are given to that effect.

Distilled water should be habitually used in dispensing.

## LECTURE XXI.

### READING AUTOGRAPH PRESCRIPTIONS.

It has been my custom in previous sessions to show you a few autograph prescriptions, and make a few brief remarks upon them, in my lecture on Dispensing Mixtures ; but the time that could in that way be devoted to it was much too short, considering the importance of the subject, and the great interest which has been shown in the study of such collections. I therefore now propose to devote an entire lecture to the reading of autographs.

The selection to which I shall direct your attention has been made from my small collection, mainly with the view of exemplifying the modes of clearing up ambiguities of different natures. Some have been added simply as exercises in reading diverse handwritings. A facility in this art is only acquired by practice, and is often found wanting in the candidates for examination who go from situations where little dispensing is done, or where the prescriptions are almost exclusively from the hands of two or three practitioners.

Those who habitually see a diversity of styles will scarcely ever meet with English prescriptions which they cannot read. My selection does not contain the most difficult prescriptions which I have met with, which have usually been German, and would have proved less instructive and less practically useful than those which are simpler in their nature ; and being indigenous to our soil, will more fairly represent what every dispenser may expect to meet with, and should be prepared to read. The selection is made entirely from prescrip-

tions which have been brought to me in the usual course of trade, and is only a small percentage of the number of those which might have afforded interesting points for comment. Patients usually object to parting with their autographs, and I have been limited to such as have been left in my hands generally by accident, sometimes by permission, and a few tracings which I have made with every care when I could not retain the originals.

As a matter of course, the superscriptions and subscriptions have been withheld, but in other respects the lithographer was instructed to copy them with every possible fidelity, neither adding nor subtracting any mark or spot which could influence for better or worse the intelligibility of the originals, and he has accomplished his task with remarkable exactitude.

From my familiarity with the selection, and many more of a similar character, the difficulties to me have in most cases entirely disappeared, and I feel that some apology may be needed, to the more skilful among my readers, for presenting such as will scarcely tax their skill ; but, on the other hand, my chief object is to supply a little useful experience to those who are deficient in it, and this object I trust is not inefficiently promoted, considering the space at my disposal. I advise the student reading these pages to examine the fac-similies carefully one at a time, as he would a prescription presented for dispensing, to make a copy, and write notes of any points which appear to require comment ; when this is done, *and not till then*, to read what I have said upon the same formula, and draw another lesson from it by a comparison of his notes with mine. A few of the fac-similies are given as further illustrations of style, without involving such obscurities as call for special comment ; of these I have simply given my reading.

I do not offer any comments upon prescriptions rendered intentionally obscure with the view of their being dispensed at some particular establishment. I am happy to say I have





2  
Please to keep this prescription & not  
send a copy of it to Mr. B

R. Quina disulphatis gr. xxx  
intilla e limone recente quantum  
sufficit Succi ad quoniam  
salvandam et adde

Aqua 3x

Stychnie gr 1

M. fiat mistura cuius  
sumat coch: 1 maximum  
bis die, circa 11<sup>ma</sup> a. m.  
et 4<sup>ta</sup> hora p. m.

March 21 / 60

very rarely met with such, and do not think them worthy of notice. Of those which are intended for general reading, a very small percentage, not more than one or two in a thousand, give real trouble to an experienced dispenser, a proportion by no means large, considering the circumstances under which medical men are frequently compelled to write. Few men have the faculty of writing well when hurried. Under these circumstances, the characteristics which always mark a mature handwriting may become so exaggerated as to render it ambiguous to one unacquainted with the usual style of the writer, while those who are acquainted with his caligraphy would experience no difficulty.

No. 1.

Master D.

R	Linimenti Opii . . . . .	℥vi.
	Linimenti Camphoræ Comp. . . . .	℥ii.

M. Ft. Lin.

“Sign ” For external use only.

Oct. xxiv. 63.

Many of the letters in this prescription are badly formed, but as the words are written in full, there can scarcely be said to be any difficulty in reading it. Physicians who take the trouble to write their words in full scarcely ever give the dispenser room for a misunderstanding.

No. 2.

Please to keep this prescription, and not give a copy of it to Mr B.

R Quinæ disulphatis . . . . . gr. xxx.  
 instille e limone recente quantum sufficit succi ad quinam sol-  
 vendam et adde,—

Aquæ . . . . .	℥x.
Strychniæ . . . . .	gr. i.

M. fiat mistura cujus sumat cochl. i. maximum bis die circa  
 11mâ A.M. et 4tâ horâ P.M.

No. 2 is a well written, and at the same time an interesting prescription, written by a physician of high standing for a chemist of equally high standing, the latter being kept in the

dark as to the nature of the medicine he was taking; the physician's instruction being, that the patient was not to have a copy of the recipe; the patient supposed himself to be taking some simple bitter. There is a chemical oversight on the part of the physician; though he carefully directs lemon-juice to be dropped upon the quinine in a quantity sufficient to dissolve it; he then simply says, add 10 ounces of water and 1 grain of strychnine. A drug like strychnine, which is very powerful and not very soluble, should have had more explicit instructions to insure solution, or otherwise a careful distribution of the doses. Had the care of the dispenser not been greater than that of the prescriber in this particular, the patient might easily have had a serious overdose when he came to the bottom of his bottle. Ordinary silver tablespoons of the present day usually hold about one fluid ounce; if the mixture were made to measure 10 oz. or 11 oz., and the patient took such a tablespoonful for a dose, he would be taking rather more than the maximum dose according to the Pharmacopœia. As the physician uses the superlative degree in his designation of spoonful, this might naturally have been supposed; at any rate, it was not considered necessary to caution the patient to use a graduated measure, and he, supposing that he was only taking "bitters," carried the bottle in his pocket and took the medicine by mouthfuls, until physician and patient were both roused to a sense of danger by symptoms of overdosing with strychnine making their appearance.

In this case, the solution of the strychnine, as well as of the quinine, should have been ordered; the exact measure of the mixture should have been indicated; the dose should have been unequivocally described as  $\text{ʒss.}$  or  $\text{ʒj.}$ , according to the intention, and the patient should have been instructed to use a graduated glass or other accurate mode of estimating the quantity; the latter precaution is always desirable, and should never be omitted when a full dose of a powerful drug is ordered.



Prof: Saldaña } V 14  
 Herat: amonon

3ii

Well: Rosam } 3

~~not collected~~

not gangusma

Utatur Saepae  
 ue

Servant of the  
 Anderson



Mr. H

2

R. Est Alon Co  
Est Hrosey  
Pu Khai Comp an gr ip ~~th~~  
Hui om note Cap  
Mitte XIV

R. Die Tarsaxi  
Inf Gen Comp an 3 VII  
Soda Bicarb 3 1/2  
Bismuth Tris nit 3 II ~~th~~  
Cabl 3 I bis dei

bet 16/64 R. ol Croton ol olive  
Gm. Trub an 3 I ~~th~~ foli  
om note aff



No. 3.

Inf. Salveae	.	.	.	.	3̄viii.
Muriat. ammon.	.	.	.	.	3̄ii.
Mell. rosarum	.	.	.	.	3̄iss.

(M. ft. collut. oris [erased]).

M. ft. gargarisma utatur saepe de die.

Servant of Mr Anderson.

The first line in this prescription may give a little trouble to any one not acquainted with the handwriting. This arises simply from a little peculiarity in the form of the "v," and is cleared up by comparing the same letter in the word "Servant" and in "Lavand." occurring in No. 12. The "ss" following "Mell rosarum" should also be noted, being a little different from the usual form of this symbol, and may aid the interpretation of any similar mark when met with. The remainder will give no difficulty.

No. 4.

R Ext. Aloes Co.

Ext. Hyosecy.

Pil. Rhei. Comp. . . . . āā gr. iſs.

M. ft. Pil. om. nocte Cap.

Mitte xiv.

R Dec. Taraxaci

Inf. Gent. Comp. . . . . aa 3̄viii.

Sodæ Bicarb. . . . . 3̄ſs

Bismuth Tris. Nit. . . . . 3̄ii.

Capt. 3̄i. bis. die.

R	Ol. Croton.	Ol. Olivæ	.	.	.	} āā 1 dr.
	Spt. Tereb.	.	.	.	.	

M. ft. Lin. om. nocte affr.

Oct. 16, 67.

We could scarcely find a fault with the writing of this recipe; the only words about which a tyro could hesitate are "Tris." in the bismuth, the "Spt. tereb," and "Affr." But having mastered these, we are still in doubt as to the intention of the first line. What is compound extract of aloes? "Pil. aloes, co." and "Ext. aloes aq." are two readings which

naturally suggest themselves in the absence of any known formula for a compound extract of aloes. The former of these made as it used to be with aloes and extract of gentian, was judged the most probable, and as it was at any rate a safe interpretation, it was acted upon without further ceremony.

## No. 5.

R	Ferri Biphosph.	.	.	.	gr. xx.
	Sacchar alb.	.	.	.	ʒii.
	Aquæ	.	.	.	ʒii.
	Cochl. i M. bis die.				

The adjective qualifying "cochl." is, no doubt, "m.;" but whether it is min., med., or mag., we are left at our discretion to determine. The mixture being prescribed for a child, and in quantity of only two ounces, a teaspoonful was the translation fixed upon.

## No. 6.

Mrs G.

R	Pulv. Opii. comp.	.	.	.	gr. x.
	ft. Pulv. post sing <sup>s</sup> deject Liquid sum.				
					Mitte vi.

Aug. xiii.

This illustrates the difficulty which is sometimes found in reading good writing, if it be folded before it is dry—a difficulty for which the patient may be as much to blame as the prescriber.

It also illustrates the danger which may arise from repeating the use of old prescriptions; the dose indicated in the Pharmacopœia for compound powder of opium is 2 to 5 grains; and though 10 grains only contains 1 of opium, it is a larger dose than we would expect to be given with the simple instruction, "after each loose motion." The fact is, at the time it was written (1859) there was no official compound powder of opium; our knowledge of the prescriber's habit led us to supply compound chalk powder with opium, which subsequent inquiry proved to be his intention.

Mr S

11

By Dale Birchard. It  
 has been ~~discussed~~ <sup>discussed</sup> in  
 Legum. Ann. Quilley  
 Aug. 1111





2

My Dear Tarapaci  
p. XXXXVI  
Fello Bob. Jospia  
p. XXIV

h. H. pil. XII  
esp. 1 hi chi

~~Bob~~ Jospia  
June 29 1863.





Mix &

℞ Extr. Aloes aquos gr  $\frac{1}{4}$   
 Ferri Sulphat gr  $\frac{1}{4}$   
 Extr. Hyoscyam  
 — Gentian. comp ii

~~℞~~ ~~℞~~  
 Pilula semel vel bis die  
 capienda \* xv

Die Aprilis 23 1856

+ at meal times

No. 7.

R	Extr. Taraxaci	.	.	.	gr. xxxvi.
	Fell. bov. inspiss.	.	.	.	gr. xxiv.
M.	ft. pil. xii.	Capt. 1 ter die.			

Mrs O. B.

June 29, 1863.

No. 8.

Miss E.

R	Extr. Aloes aquos	.	.	.	gr. $\frac{1}{4}$ .
	Ferri Sulphat.	.	.	.	gr. $\frac{1}{4}$ .
	Extr. Hyoscyami				
	Gentian	.	.	.	āā gr. ii.
	ft. Pilula semel vel bis die capienda × xii.				

Die Aprilis 23, 1856.

× at meal times.

This may be taken as a specimen of good writing. As it came from the hand of the physician, it probably did not present the shadow of an ambiguity. The one point which requires comment is the mark × which follows "capienda." It was there, and not crossed out, when first the prescription came under my notice; and when, after an interval of some months, during which it had been dispensed in several other towns, it was presented again, the × was crossed out with pencil, and a pencil mark xii. was placed after it, indicating that some dispenser had interpreted the × as ten. We can scarcely suppose that he read it ten pills once or twice a day, but probably supposed that the physician intended "Mitte ×," and the dispenser had preferred to make 12 as a more convenient multiple of the quarter grains. But a little consideration will show that the × is intended for \*, referring to the footnote—"× at meal times." This, as it appears to be in another handwriting, has probably been added by the patient, who was no doubt quite unconscious that adding × after a word might have multiplied her dose by ten.

## No. 9.

R	Extr. Belladon.	.	.	.	gr. iss.
	Butter of Cocoa	.	.	.	q. s.
M.	ft. supposit., p. r. n. utend	.	.	.	viii.
R	Acid. Carbolic	.	.	.	℥ss.
	Glycerin	.	.	.	℥iiss.
M.	Use 10 drops.				

## No. 10.

Hab. Tinct. Iodin. C. . . . . ℥iv.

Sig. The Iodine paint for external application.

July 13, —68.

## No. 11.

Mrs S. Baby.

R	Hydr. c. Creta	.	.	.	gr. i.
	Pul. Cretæ co.	.	.	.	gr. ii.
M.	ft. Pul. omni nocte sum.				Mitte xii.

June 7, —57.

Nos. 9, 10, and 11 do not call for any particular remarks.

## No. 12.

R	Spirit lavend. comp.	.	.	.	℥iv.
	Spirit nitri dule.	.	.	.	℥ii.
	Laudani	.	.	.	℥ii.
M.	Sumat bis de die 20 guttas.				

This autograph is useful as enabling us to compare the “v” in “Lavend.” with the more doubtful “v” in “Salviæ,” in No. 3, by the same writer.

## No. 13.

R	Tr. Ferri Muriatis	.	.	.	℥iii.
	Lq. Arsenicalis	.	.	.	℥iss.
	Magnes. Sulphatis	.	.	.	℥i.
	Aquæ	.	.	.	℥viii.

℥ss. t. d.

R Zinci Ben.

Here we have an instance in which the patient afforded all the information which a careful examination of the writing did not supply. The whole prescription was evidently hurriedly

N 41 Bellamy  $\frac{7}{10}$

Baller & Calver  
Wm H. Jones

per altered

very

N 42 Calver & Co. Sp

Shore

~~July 10~~ ~~Nov 10~~



Wm. C.

Pat. Hie: Edwin: C. 314

of the Edwin: C. Hie:

for external application

Aug 13/68.





2

Mr S Bulz

of Apr. 1. 1871  
 Pul. 1871

to Mr. Wm. W. W.

Sum. 1871

Jan 1/71 11/6/56



12 Spirit Ladani comp  
311  
Spirit nitro dull  
311  
Ladani 311

m

Spirit bis aa aa  
20 guttas



} 4 hours Transits  $\frac{3}{4}$   
 } Annals  $\frac{3}{4}$   
 } Major Sclerites  $\frac{3}{4}$   
 } Aqua  $\frac{3}{4}$   
 }  $\frac{3}{4}$

} 4 hours  
 } 4 hours







N<sup>o</sup> 1

June 14

R Pulb Iovcin ꝑ vi  
 Hydrar e (rata ꝑ iv  
 in pil duas stat  
 sumendas.

R Magnes bolā 3ꝑ  
 Pulb Rhiz ꝑ xv  
 Symplic Trujā 3ꝑ  
 Trujā Trujā 5i. 44  
 Miv. Hydrargan dilut  
 Haustus Agnō — 3<sup>ii</sup>. [M. v.]  
 post horas duas  
 sumendus.

written, and at first glance appeared to threaten some trouble ; but the first word was evidently "Tr.," and "Ferri muriatis" followed without trouble. The first letter in the second line appeared to be L, followed by a letter below the line ; "Lq" appeared probable, and "Arsenicalis" suggested itself as the most likely liquor in this combination and dose.

The first letters look more like "Al" than anything else, but there is sufficient resemblance between the second and the "rr" in "Ferri" to justify its being read as "r," and if this be accepted there need be no hesitation about the remainder of the word. "Magnesiæ sulphatis ʒj," and "Aqu. ʒviij," are as clear as need to be. The directions "ʒss. t.d." also need not give any trouble. But by the time the mixture was ready for the hand of the patient, who was waiting, I had not made up my mind how to read the last line. The mixture suggested a skin disease, and this pointed to zinc ointment as being the most probable interpretation of the last line ; but the matter was not so certain as to let me feel comfortable in supplying it with the chance of its being wrong. I therefore ventured to ask the patient if the doctor had instructed him with regard to the use of ointment. To which he replied, "Yes, he told me how to use it," and then I supplied a pot of zinc ointment without the doctor having been troubled, or the patient knowing that I had felt any hesitation about it.

No. 14.

Mrs C.

R	Pulv. Doveri . . . . .	gr. vi.
	Hydrarg. c Creta . . . . .	gr. iv.

In pil duas statim sumendus.

R	Magnes. Ustæ . . . . .	ʒss.
	Pulv. Rhei . . . . .	gr. xv.
	Syrupi Zingib. . . . .	ʒss.
	Tinct. Zingib. . . . .	M. xx.
	Acid Hydrocyan. Dilut. . . . .	M. v.
	Aquæ . . . . .	ʒii.

Haust post horas duas sumendus.

The word "Ustæ" is not very clearly written, and not

being in constant use now, as indicative of calcined, may give trouble to some of our younger friends; the last few lines become somewhat confused for want of space, but should give no trouble if attacked in detail.

## No. 15.

The Rev. Ernest T.

R	Hydr. Bichlor.	.	.	.	.	gr. i.
	T. Opii	.	.	.	.	ʒss.
	Inf. Quass. conc.	.	.	.	.	ʒi.
	Aquæ	.	.	.	.	ʒi.

Cochl. 1 min. ter die. ex aqua.

R	Pulv. (?)	.	.	.	.	gr. vi.
	Bismuth	.	.	.	.	gr. iv.
ft Pulv. o. n.		.	.	.	Mitte vi.	

In the first line of this formula the quantity is undoubtedly one gr., though the symbol would equally well represent ʒj. and we are guided entirely by the dose which would be appropriate. The consideration of doses constantly comes to our aid in clearing up difficulties, not only in regard to the interpretation of ambiguous quantities, but frequently in cases where the drug itself or the special preparation of a drug would, in the absence of this aid, be a matter of doubt. Take for example the next line, the drug is opium, and the quantity half a drachm, for sixteen doses, one thrice a-day. Now the prefix to "Opii" looks more like P than any other letter in the alphabet, but this would make the dose nearly two grains of opium thrice a day, a very unlikely dose, especially associated with an ordinary alterative, and comparing the doubtful letter with T in "The" and the initial of the patient's name (which, however, looked more like Jones than anything else, until in reply to my inquiry, he gave a name beginning with T.); thus all things considered, there was no doubt left that "Tinct. opii" was the intention, and this is supported by the notable difference in the mode of writing "Pulv." in the second half of the prescription.

The Rev. {rout. 1}

1) Hrd Butler 28

1.000 3/4

Ref Luf ~~the~~ come 3/4

Yall 1 <sup>as a 3/4</sup> <sup>ten</sup> <sup>lie</sup>

en aya"

1) Pub bar 1 1/2

to is m. 1/2 10

1) Pub 1. 1/2

with 1/2 16970











D.

25

Mrs J

By Anti-Slavery Socy

By Mrs Smith

With Amount £ 50

Mr General Pitt

his share of the

being up to

in Nov 22 1852

$$\begin{array}{r} 1/3 \\ 1/3 \\ 1/3 \\ \hline 1 \end{array}$$

The first article in the formula for powders is probably one of the most difficult to interpret with which I have met. It has been translated, "Rhei," "Tragacan.," "Mag.," "Doveri," "Gum.," "Ferri.," "Arom.," "Cinn.," "Cret.," &c., all of which may have something said in their favour, but none of which can be definitely fixed upon by any internal evidence. Any one familiar with the handwriting might perhaps read it without difficulty.

No. 16.

R	Ol. juniperi	.	.	.	.	ʒi.
	Axung	.	.	.	.	ʒi.
M	* * *	S. for rubbing in.				
		for M. C.				
	Nu	* * *	Hotel			
	11-6-57				* * * *	

There are several points in this which I failed to solve satisfactorily, but which I thought of little moment, as there appeared no ambiguity about the ointment or directions. The first illegible word no doubt means mix, but what is the precise writing I am not prepared to say; the second appeared to be the name of a hotel, and the third the signature, so the ointment was made without any hesitation; but the patient said my production was quite different from what he had in Germany, the latter being brown and smelling like tar. No doubt oil of juniper tar was the intention, and sufficiently well understood in the prescriber's own neighbourhood.

No. 17.

Mrs D.

R	Acid. Nitrici.	.	.	.	.	ʒiii.
	Syr. Simpl.					
	Tinct. Aurantii.	.	.	.	.	āā ʒii.
M.	Capt. cochl. Th. 1	bis die ex cyath	vinos aquæ.			

Die Nov. xx. 1852.

The greatest difficulty here is to read the words "Cap. cochl.," the want of clearness arising mainly from the two words being run into one. The dose may be taken for a teaspoonful if the letters indicating it be "Th.," but it is quite

possibly intended for M., in which case the dose may be a teaspoonful, a dessert spoonful, or a table spoonful, the first is the most suitable dose; the dose of strong nitric acid being about two to five minims, the dose in the prescription being about six minims if the mixture is made to measure four oz., or little more than five minims if made to measure four oz. and three drachms. This appeared sufficiently within ordinary experience to justify the mixture being made without writing to the physician at a distance to have it confirmed; but to the patient's alarm, the cork was blown out of the bottle with the evolution of nitrous fumes, and probably the conversion of part of the sugar into oxalic acid. If diluted nitric acid were intended, I would alter my reading of the directions thus, "Cap. cochl. mag. i. bis die," &c.

## No. 18.

R.	Carb. Ferri, Sacch.	.	.	.	gr. viii.
	Pulv. Aromat.	.	.	.	gr. ii.
	Carb. Sodæ Exsicc.	.	.	.	gr. iv.
M.	ft. Pulv.	.	.	.	Doses xii.
Sig.	Stomachic Powders. one in a little gruel twice day.				

This was written by one physician for another, the patient bringing it to us with the declaration that he could not read it, but all the difficulty disappears after a careful examination. In the second line, the letters are evidently not exactly as I have rendered them, but we have only to suppose that it was contracted as "Aromt." It is interesting to note, that in almost every case, the chief difficulty has been in reading the directions which are written in English.

## No. 19.

R.	Pilul. Hydrarg	.	.	.	.	Ḑ
	„ rhei co.	.	.	.	.	Ḑii.
M.	ft. Pilul. xii.					
Capiat	ii. H. s. p. r. n.					

. . . . .

R. Carb. From  
 South from  
 Vij

From ~~South~~ South from Vij

Carl South of Vij  
 from

114

From from Vij

Ly. Schmacke from

from - on in a little

from from a day



Mrs W

℞ Pilul Hydrag ʒi  
— Rhoei co ʒii ℥

℞ Pilul XII

Capit ii ℥ss, ʒ. m.

℞ Magna Carb  
Soda Carb  
Magnes Carb an ʒi  
Aqua ʒviij  
Vin Specie ʒi  
Vin Lingib ʒii ℥

Capit coctel 10<sup>th</sup> man post  
Mileas.

Die Aug 26 1854







1/3 Charcoal gas 120  
 1/3 Gas Sumial \$3 180  
 Syrup Auriantin 3/4  
 Arg. ar 3/4  
 Capt 3/4  
 M. 3/4  
 3/4 Hydrargyri 3/4  
 3/4 Belladonna 3/4  
 3/4 Morone 3/4  
 3/4 Capt. 3/4  
 3/4 pilat. 3/4

R	Magnes. Carb.				
	Sodæ Carb.				
	Magnes. Carb.	.	.	.	aa ʒi.
	Aquæ	.	.	.	ʒviiss.
	Vin Ipecac.	.	.	.	ʒi.
	Tinct. Zingib.	.	.	.	ʒii.

M. Capiat cochil. ii., mane post Pilulas.

Die Aug. 26, 1854.

No. 19 is, upon the whole, a well-written autograph, but there is a trifling error in the mixture, carbonate of magnesia being written twice; and the question arises whether it is simply an accidental repetition, or whether one of them was intended to be some other ingredient. A knowledge that the prescriber frequently ordered the carbonates of potass, soda, and magnesia in the same mixture, led to the conclusion that that was his intention. Then, as regards the dose, the figure is not very clear, it might be ii., iii., or iv. spoonfuls; there are three dots, which points to three being the number intended; but there are also three dots over 1 drachm in the quantity of "Vin ipecac." The three dots in the latter case certainly occur by some accident, and the same may be the case in the former number. Folding before the ink was dry may account for both. It appeared that the middle dot in the dose and the curling back of the tail of the latter figure in the dose, had both occurred in such a manner. Neither interpretation would make the mixture last exactly the same time as the pills. Two was ultimately decided upon, and is confirmed by the appearance of the figure when the middle dot and middle stroke are covered by a white thread.

#### No. 20.

R	Chloral	.	.	.	grs. 120.
	Potass. Bromid.	.	.	.	grs. 180.
	Syrup. Aurantii	.	.	.	ʒss.
	Aq. ad.	.	.	.	ʒvi.

M. Capt. ʒi. omni nocte.

R	Hydrarg. Bi. Chlor.	.	.	.	gr. ii.
	Extr. Belladon.	.	.	.	gr. vi.
	Conf. Rosæ,	.	.	.	q. s.

ut fiant pil. xii. Capt. 1 bis die.

The official dose of perchloride of mercury is  $\frac{1}{16}$  to  $\frac{1}{8}$  of a grain; this being  $\frac{1}{6}$  of a grain twice a-day, seems a larger dose, but a comparison with other prescriptions from the same physician showed that he was not unaccustomed to giving a full dose of this remedy; and the clearness with which it is written excluded any other interpretation. The "ss." after the ounce mark on the third line is not equally clear, but may be compared with another autograph by the same hand, No. 31.

## No. 21.

Hyd. Bichlorid.	.	.	.	gr. $\frac{1}{2}$ .
Tinct. Cinch. Co.	.	.	.	$\overline{3}$ i.
Aquæ	.	.	.	$\overline{3}$ i.

Cochl. Min. bis die ex aquâ.

In this case the quantity in the first line might be taken for half a scruple, were it not that the consideration of a suitable dose clearly indicates that half a grain must be the intention.

## No. 22.

R	Ext. Elateri	.	.	.	gr. $\frac{1}{6}$ .
	Gum Opii	.	.	.	gr. $\frac{1}{4}$ .
ft. Pil.					
Om Noct.					

If difficulty were experienced in reading the first line of this formula, an appeal to the list of extracts in the index of the Pharmacopœia would not afford us the assistance which it usually does; but a comparison of the initial of the doubtful word with the "E" in "Ext.," will at once solve the problem, and the suitability of the dose puts it beyond all question.

In this case the second word, if isolated, might give some trouble to decipher, but there is no difficulty in deciding that the first word which follows the sign R is Ext., and this guides us to the determination that the first letter of the second word is E, and then the others follow without any difficulty, "lateri." Had this not suggested itself as an evident reading, we would naturally have looked over the list of

4zd. Buller:  $\frac{1}{2}$   
Tint: Quink:  $\frac{1}{2}$ :  $\frac{1}{2}$

Aqua  $\frac{1}{2}$   
Celli in his  
crava



I Jhr. Platen gte  
 Penn oprij gte  
 Al Pil  
 om hoef







By Other Chronic  
Common C. in 1st 4th  
and C. 2nd  
Ag. Test. 3rd

1st 2nd 3rd 4th 5th 6th 7th 8th 9th 10th 11th 12th 13th 14th 15th 16th 17th 18th 19th 20th 21st 22nd 23rd 24th 25th 26th 27th 28th 29th 30th 31st 32nd 33rd 34th 35th 36th 37th 38th 39th 40th 41st 42nd 43rd 44th 45th 46th 47th 48th 49th 50th 51st 52nd 53rd 54th 55th 56th 57th 58th 59th 60th 61st 62nd 63rd 64th 65th 66th 67th 68th 69th 70th 71st 72nd 73rd 74th 75th 76th 77th 78th 79th 80th 81st 82nd 83rd 84th 85th 86th 87th 88th 89th 90th 91st 92nd 93rd 94th 95th 96th 97th 98th 99th 100th



R. Aloes Barb- gr XXIV  
and sulph. fort ~~3ij~~ 3j  
in ~~the~~ divide in pil 3j  
Two to be taken every four  
hours until the bowels are  
relieved —

R. Senna fol 3iW  
mag sulph 3ip —  
Lycopi Rhamni  
Funct Jalappa aa 3ij  
~~Must~~ Guip 3ij  
~~Ague~~ Infuse in  
agua 3vj —  
3ip 2or; hori done  
all usport



M<sup>rs</sup> I  
 K potaf. Iodid  $\frac{1}{2}$  3i  
 Ferr Am. cit 3ij  
 aqua w<sup>3</sup>viij m<sup>3</sup>  
 Mist cyus  $\frac{1}{2}$  3j ter die

extracts to call to mind any which would be appropriately prescribed in  $\frac{1}{6}$  grain doses, and with the name of which the ambiguous word could be supposed to coincide.

No. 23.

R	Æther. Chloric.				
	Spt. Ammon. Co.	.	.	.	aa gtt. xl.
	Tr. Card. Co.	.	.	.	℥ii.
	Aq. Font.	.	.	.	℥i.
M.					

Note that the quantity of the two first ingredients is gtt. xl. and not 14. It is not good practice to use both Arabic and Roman numerals in the same prescription, but we have so many examples of that irregularity, that we can never take it for granted that they will not occur in juxtaposition.

No. 24.

R	Aloes Barb.	.	.	.	gr. xxiv.
	Acid Sulph. fort.	.	.	.	M. vi.
M.	divide in pil.				vi.

Two to be taken every four hours until the bowels are relieved.

R	Sennæ fol.	.	.	.	℥iv.
	Mag. Sulph.	.	.	.	℥iiss.
	Syrup Rhamni				
	Tinct. Jalapæ	.	.	.	āā ℥vi.
	Tinct. Zingib.	.	.	.	℥ii.
	Infuse in aquæ	.	.	.	℥vi.

℥iiss. 2 dis horis donec alv. respondt.

The use of oil of vitriol as an adjunct to Barbadoes aloes can scarcely be considered elegant pharmacy, and is now fortunately of rare occurrence.

No. 25.

Mrs F.

R	Potass Iodid.	.	.	.	gtt. ℥i.
	Ferri Am. Cit.	.	.	.	℥iiss.
	Aquæ ad.	.	.	.	℥viii.

M. ft. Mist. cujus St. ℥ss. ter die.

The "gtt." preceding  $\text{ʒi.}$  in the first line is evidently a lapsus occurring, as these things frequently do, in consequence of the patient talking to the doctor all the time he is writing. It leads to no difficulty, and only needs to be erased.

## No. 26.

R	Argenti Nit.	.	.	.	gr. iiii.
	Aquæ Distill.	.	.	.	$\text{ʒi.}$
F.	Lotio. Assid. app.				

At first glance the word following aqua was supposed to be "Fortiss," which it certainly resembles, but as it is to form a lotion to be applied assiduously, the word "distill" was at once fixed upon.

## No. 27.

R	Liq. Plumb. Diacet.	.	.	$\text{ʒss.}$
	Acid Hydrocyan. Sch.			$\text{ʒi.}$
	Morph. Acetat.	.	.	gr. v.
	Aquæ ad.	.	.	$\text{ʒvi.}$
M.	ft. Lotion.	$\text{ʒss.}$	ex aqua utend.	
R	Sodæ Carb.	.	.	$\text{ʒss.}$
	Pulv. Rhei	.	.	gr. xxx.
	Tinct. Zingib.	.	.	$\text{ʒii.}$
	Spt. Ammon. Arom.	.	.	$\text{ʒi.}$
	Tinct. Cardam. Co.	.	.	$\text{ʒi.}$
	Infus. Gentian ad.	.	.	$\text{ʒviii.}$

M. Sumat.  $\text{ʒi.}$  quotidie ex aquâ.

No. 27 presents us with two or three symbols which are a little ambiguous. There might be some doubt as to whether the ounce or drachm symbol was intended in the quantity of lead solution, in the quantity of lotion to be used, and in the quantity of carbonate of soda ordered. In the latter case we find the quantity of rhubarb in the following line is written gr. xxx., and that the quantity of soda has been begun to be written in the same manner, but has been struck out, and would scarcely have been made to express the same quantity by another symbol. Then as regards the appropriateness of

R. Argenti Nit pr III  
 Aqua Potas 3i  
~~Floris~~

Amid off





} Liq Plum Tracel 3j  
 } Acid Hydrocyanic 3i  
 Morph acetat for Aqua 3vi  
 M. S. L. 3p ex aqua utenda

R Soda Carb ~~3i~~ 3p  
 Pulb Phe xxx

Pulv. Tinctura 3i

Spl. - Ammonia 3i

Muc Cardam Co 3i

Infus Gentian 3vi

M. S. L. 3i sedendi

q aqua -

R Potus Bromid. 3p

Pulb. M. S. L. 3p  
 of water & take two  
conglutins every night

an ind. rubber ball syringe





Sept. 21. 72

L. F.

A Bismuth Albi  
 3ij  
 Tinct Card Co

3℥ Acid Inset  
 Mus Fr. a a m m XVI  
 Infus Osmunda  
 ad 3viii m 81 3℥  
 ter de e aqua

A Pil Col Co XII  
 8j - 3 p. r. m.

the two problematical quantities, the eighth part of half an ounce is a more usual dose than the eighth of half a drachm, and we conclude to read it  $\text{ʒss}$ .

The question of appropriateness in the other two ambiguous quantities will also lead us to conclude that they also are both intended for  $\text{ʒss}$ .

The word preceding "Zingib." appears to have been written "Pulv." first, and afterwards "Tinct." This is confirmed by the fact that it is more usual to order  $\text{ʒij}$ . of tincture of ginger in an eight-ounce mixture, than the same quantity of powdered ginger.

No. 28.

R	Bismuth Albi.	.	.	.	$\text{ʒjss}$ .
	Tinct. Card. Co.	.	.	.	$\text{ʒss}$ .
	Acid. Nit. et Mur. Dil.	.	.	.	$\text{āā min. xvj}$ .
	Infus. Diosmæ, ad.	.	.	.	$\text{ʒviij}$ .
M.	St. $\text{ʒss}$ . ter die, ex. aquâ.				
R	Pil. Col. Co., xij.				

St. i. p. r. n.

This autograph has more the appearance of confusion, from the lines not being divided in the usual manner. The word "Nit." is the most difficult to read, and this the more so from the want of a dot to the "i," and a cross to the "t"; but the following word "Mur." being clear enough, naturally leads to the correct interpretation, and we observe that various "i's" and "t's" are without their usual appendage. I felt no hesitation in reading the infusion as "Diosmæ," though the patient said it was not the same as he had had previously. The prescriber being at a distance, I discussed the possible readings of the word with the patient himself, and satisfied him of my correctness. We could only conclude that some other dispenser had put a different interpretation upon it, which he admitted it was quite possible to do. "Inf. quassiæ" seems the next in order of probability. It is always a comfort in a case of this kind to be able to assure patient that the alternative is not a dangerous one.

## No. 29.

R	Bismuth Trisnit.	.	.	.	℥jss.
	— Liquor	.	.	.	℥ss.
	Glycerin	.	.	.	℥ss.
	Liquor. Potas.	.	.	.	℥ij.
	Chlorodyne	.	.	.	℥j.
	Acid. Hydrocy. dil.	.	.	.	℥50.
	Ext. Belladonnæ	.	.	.	gr. vj.
	Tr. Calumb.	.	.	.	℥jss.
	Inf. —	.	.	.	ad. ℥x. M.
	℥ss. ter in die. Sd.				

Emp. Belladonnæ Co.

5 inches × 4, margin adhesive inclusive.

Oct. 21/71.

## No. 30.

R	Magnes. Sulph.	.	.	.	℥iv.
	Acid. Sulph. Dil.	.	.	.	℥jss.
	Syr. Zingib.	.	.	.	℥ss.
	Tinct. Aurantii	.	.	.	℥iij.
	Inf. Aurantii Co.	.	.	.	ad. ℥viiij.

M. Capt. ℥j. ter die.

Dec. 4, 1872.

Dec. 9, 1872.

Rep. Mista. cum Magnesiae Sulphatis ℥iv. ad. ℥viiij.

This may be considered a well-written prescription, with the exception of one word, viz., “Zingib.,” of which the letters “z” and “g” are the two wanting in clearness. The initial does not occur in any other word in the prescription, and we have not the advantage of making a comparison; but as the preceding word “Syr.” is clear enough, and we have only to consider the list of syrups to see at once that zingib. is the one which fits the occasion; and this may be confirmed if necessary by comparing the “g” with the same letter in the word *magnesia*, which occurs twice.

R. Bismuthi Trisulph 3ij

— liquor 3j

Glycerum 3j

liquor Potas 3ij

Chlorodyne 3j

Aqua Hydrag. dil. M 50

Ext. Belladonna 3ij

L. Celv. 3ij

Hy. — ad 3℥

3j in die P.

Emp. Belladonna (c.

5 mils x 4. M. aq. in. ad. in.

inclusion

Oct 21/71.





for Mr L

R: Magnes. Sulph 3iv

Acid. Sulph Lix 3ip

Syr. Equis 3p

Tinct Aurantii 3iij

Inf Aurantii Co ad 3viii

As per 3i to be.

4 Dec 1872

Dec 9. 1872

Re Smith's cum magnesias

Sulphates 3iv ad 3viii





Chlorophormi in x x

Magnus Carl M. M.

Magnus Carl M. M.

Magnus Carl M. M.

Magnus Carl M. M.

Magnus Carl M. M.

Magnus Carl M. M.

Magnus Carl M. M.

Magnus Carl M. M.



Mip E.

R Hyd Chlor  $\text{gr} \times \times$   
 Iric Hyocy 3i  
 Aqua 3i + M.

(Draught to be taken at  
 bed time.)

Nov 9/72

No. 31.

	Chloroformi	.	.	.	.	℥ <sub>xx</sub> .
R	Magnes. Carb.	.	.	.	.	ʒj.
	Magnes. Sulph.	.	.	.	.	ʒss.
	Pulv. Rhei.	.	.	.	.	ʒij.
	Pulv. Zingib.	.	.	.	.	ʒss.
	Liq. Morphine Mur.	.	.	.	.	ʒij.
	Inf. Gentian	.	.	.	.	ad. ʒviij.

M. Capt. ʒss. ter die.

No. 31 presents a good example of how one part of a prescription throws light upon the ambiguity of another. There is no difficulty in any part except in the quantity of two ingredients and the dose. We might suppose the quantity of sulphate of magnesia to be ʒj., if it were not for the context; and, under similar circumstances, we would say the quantity of powdered ginger was intended for ʒss. But the dose shows the necessity of our carefully considering the intention of the whole of these figures. We notice in every case where we are sure the figure 1 is intended that the dot over it is not omitted, that the figure in the quantity of carbonate of magnesia differs more from that in the quantity of sulphate of magnesia than the latter does from that indicating the dose, and that the half drachm of powdered ginger is indicated by one "s," not two; taking these circumstances all into account, we conclude that all the three figures are intended as abbreviations of the fraction "ss," and the reading of the recipe is decided as above.

No. 32.

Miss E.

R	Hydr. Chlor.	.	.	.	.	gr. xx.
	Tinc. Hyoscy.	.	.	.	.	ʒj.
	Aquæ	.	.	.	.	ʒix. M.

Draught to be taken at bedtime.

Nov. 9/72.

We have here to note the ambiguity resulting from abbreviations.



viating the name of a drug. For many years the translation of "Hydr. chlor." has been chloride of mercury, and the only doubt would have been *which* chloride. But neither of these could be intended in this case, and we find the true interpretation in hydrate of chloral. The dose, the mode of administration, the association with tincture of henbane, and the fact that it is a night draught, all confirm this reading. But it will be necessary to remember in future, where we have not these clear indications from the context, that "Hydr. chlor." may be either calomel, corrosive sublimate, or hydrate of chloral.

## LECTURE XXII.

### PILLS—POWDERS.

Pills—Physical Requirements—Excipients for Rhubarb—Aloes—Creasote—Camphor—Essential Oil and Aqueous Extract—Hygroscopic Extracts—Copaiba—Sulphate of Iron—Oxide of Silver—Carbolic Acid—Cutting, Finishing, Coating, Silvering. Powders—Mixing, Dividing, Packing.

#### *Dispensing Pills.*

THE object of making any drug into pills is to have it in a form which is at once portable, permanent, and divided into convenient doses which are not nauseous to take.

The skill of the dispenser will be shown in the uniformity of his success in obtaining these results with the very various materials which he is expected to make into pills.

It is usual for the physician to prescribe the active constituents of the pill, and the dispenser to add what excipients he may judge desirable, and the selection of a suitable excipient is one point in which a good deal of judgment is required.

Pills are liable to become soft and sticky, or hard and insoluble; they sometimes become dimpled or angular, sometimes mouldy; in any of which conditions they show that the operator has failed to make them what they ought to be, though it does not necessarily follow that he is to blame in the matter; it may be that medicines have been ordered which are not suitable for this mode of administration.

But a great deal may be done even with the most intractable ingredients, if there is a systematic knowledge of the mode of proceeding. No material is plastic without the pre-

sence of some fluid or semi-fluid body. But semi-fluids are of two distinct kinds, such as mucilage of acacia and mucilage of tragacanth; the former, even when it contains a very large proportion of gum, will flow by its own weight; the latter, even when it contains a large proportion of water, will not flow unless under some further mechanical force. Tragacanth and water alone might be made into pills which would not fall out of shape, but acacia and water would be wanting in firmness unless some solid material were also present. If it were required to impart adhesiveness to a powder such as ginger, the acacia would be suitable; but if it were required to give firmness to a soft extract, such as acetic extract of colchicum, tragacanth would be more successful. If too much tragacanth be used the mass will be *too* retentive of its shape, and after having been rolled and cut, the pills will be found to retain a cylindrical form, which continues to return after they have been repeatedly rounded; but if some fibrous material had been used instead of part of the tragacanth, it would have imparted that condition which enables any form to be given to the pills, and to be permanently retained by them. As tragacanth is not readily soluble, it should only be used in small quantities unless in connection with hygroscopic or very soluble substances, otherwise the ready activity of the pill would be impaired.

We may compare the pill with the human body. A man could not stand upright if he had not a bony framework, nor could a skeleton stand without muscles and ligaments; in addition to these, the blood and juices of the body are required to impart that condition which possesses at once firmness and the power of motion.

So in the structure of a good pill, we find the analogue of each of these essentials to a perfect animal. A fibrous powder represents the bones of a pill; gum or extractive matter stands in the place of the muscular element; and the moisture, without which gum is not adhesive, represents the blood. By the junction of the three you may obtain a perfect

pill, but any two separately will fail to produce the requisite qualities.

I have now to direct your attention to prescriptions and samples of pills, illustrating deficiency of one or other of these elements, and to point out to you the appropriate excipients by which to supply the deficiency.

	No. 1.	
R Pulv. Rhei	. . . . .	ʒi.
	ft. Pil. xx.	

This may be combined with water, syrup, mucilage, treacle, glycerine, or solution of potash. Water makes a rather crumbly mass,—solution of potash the toughest. But while we may be satisfied with the physical qualities obtained by the alkaline liquor in this instance, its use might naturally be objected to as an unwarrantable addition when so many other simple excipients might have been selected with perfectly satisfactory results.

With water alone, the pills become very hard, and are only slowly dissolved; consequently there is a double advantage in the use of syrup. The mass works better, and from the free solubility of the sugar, however hard the pills become, they are always more readily acted on by water.

Samples of pills by these two forms, now twelve months old, are both very hard. Those made with syrup are the hardest, but also the most soluble; one of each being dropped into separate dishes of cold water, the pill with syrup gives a coloration within two minutes, but that made with water shows no appearance of solution in ten times that period. In the course of three hours the former pill will be completely disintegrated; the latter requires about double the time.

It is a great advantage to add precisely the right quantity of excipient at once. For one drachm of rhubarb powder, half a fluid dram of water, syrup, or glycerine may be used. If only half the required quantity be added at first, a mass is formed which is too hard to roll out, and with which it is very difficult to mix an additional quantity of the excipient.

Aloes and gum resins generally require much less excipient, thus—

No. 2.					
R	Pulv. Rhei	.	.	.	āā
	— Aloes	.	.	.	3ss.
ft. Pil. xx.					

Is conveniently made with twenty minims of syrup instead of thirty, which would have been used if the whole of the powder had been rhubarb.

If simple rhubarb pills are made up with water only, in an iron mortar, the rhubarb is blackened by the mortar; but if syrup or some other excipients be used, the blackening does not take place. This is an example of the conservative power of sugar, which is often useful both in protecting iron compounds against oxidation, and preserving vegetable matters from the injuries they are liable to sustain by long keeping.

No. 4.					
R	Extr. Aloes. Aq.	.	.	gr. xxxvj.	
	Cons. Rosæ	.	.	gr. xvij.	M. ft. Pil. xii.

No. 5.					
R	Extr. Aloes Aq.	.	.	gr. xxxvj.	
	P. Ligni .	.	.	gr. vj.	
	Glycerin	.	.	gtt. vj.	
M.	ft. Pil. xii.				

No. 6.					
	Extr. Aloes Aq.	.	.	gr. xxxvj.	
	Sapo. Hisp. (Nov.)	.	.	gr. xij.	
M.	ft. Pil. xii.				

I have experienced no difficulty with extract of aloes pills since it became the custom to use the extract in powder. When the rule was to have the extract of a pilular consistence the pills were with difficulty made to retain their shape.

The three preceding recipes show the comparative advantages of these excipients in forming aloes into pills. The con-

serve makes a good mass, which works well and does not get hard, but if not kept very dry the pills become soft and sticky, and perhaps run into a lump, at any rate do not retain their shape.

No. 5 remain soft and sticky, but do not lose their shape; and I do not know any fibrous material which will impart this retentiveness of shape to a soft pill with so small an increase of bulk as fine sifted sawdust. It may be considered "pure bone" to a pill, according to the analogy which I traced to you just now; and glycerine is the most perfect retainer of moisture, and consequently softness, which we can select; but being hygroscopic, the pills are apt to become too soft and sticky, if not kept in a very dry place; we can easily reduce this tendency by diluting the glycerine with water or spirit, used in larger or smaller proportion as circumstances indicate.

No. 6 retain their shape and solubility tolerably well if the extracts be in powder, and the hard soap be soft; but the pills become hard when old.

## No. 7.

Bismuth Trisnitr. . . . . ʒij.  
ft. Pil. xii. with Mucilage of Glycerine, ʒss.\*

These, though they contain  $12\frac{1}{2}$  grains of material in each pill, are no larger than ordinary five-grain pills. The excipient is the same I recommended when treating of official pills, and is generally useful where it is desired to form into pills any powder which is bulky, amorphous, and not adhesive.

## No. 8.

R	Creosote	.	.	.	gtt. xxiv.
	ft. Pil. xii.				
With	P. Amygdal.	.	.	.	gr. xxx.
	Glycerin.	.	.	.	gtt. xij.
Or with—					
	Cera Flav.	.	.	.	} āā gr. xij.
	P. Saponis	.	.	.	

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* R	P. G. Tragacanth	.	.	.	ʒij.
	Glycerin.	.	.	.	ʒix.
	Misce et adde				
	Aquæ	.	.	.	ʒiv.

The first of these forms depends for its efficacy upon the power of almond powder (in preference to that deprived of its oil), to facilitate a permanent union of oily and watery matters, and thus prevent the creosote "sweating out," but the pills do not readily dissolve. The latter form is preferable, as the wax converts the creosote into a jelly-like paste, and the soap enables it to emulsify in the stomach.

## No. 9.

R	Camphor	.	.	.	.	gr. xxxvj.
	ft. Pil. xvij.					
With	P. Amygdal.	.	.	.	.	gr. xij.
	Glycerine	.	.	.	.	gtt. xv.

## No. 10.

R	Camphor	.	.	.	.	℥ss.
	Ol. Caryoph.	.	.	.	.	gtt. vj.
	ft. Pil. xij.					
With	P. Saponis	.	.	.	.	gr. vj.

## No. 10B.

R	Camphor	.	.	.	.	℥j.
	ft. Pil. xij.					
With	Ol. Ricini	.	.	.	.	gtt. iv.
	P. Sapo.	.	.	.	.	gr. ij.

## No. 10c.

R	Camphor	.	.	.	.	℥j.
	Ext. Hyocyami	.	.	.	.	℥ij.
	ft. Pil. xij.					

These formulæ illustrate several modes of giving camphor in pills.

No. 9 is similar to the creosote pills just above; but as we are now dealing with a concrete oil, there is no need of a large bulk of absorbent material; the larger proportion of glycerine, and smaller quantity of almond meal, make a more soluble pill.

No. 10 gives us the task of combining both camphor and

essential oil into a pill, which is simply done by the use of powdered soap, but would have presented considerable difficulty if the quantity of oil of cloves had been so great as to dissolve the camphor.

In 10B, we purposely added oil as an excipient, and it is about the best we can use to make 5 grains of camphor into a pill of no extraordinary size. The camphor must be *rubbed* to a powder with the oil, and then *worked* into a mass with the addition of the soap.

The pills Nos. 9, 10, and 10B, being immersed in cold water, we find the first is readily disintegrated, the second requires several hours, and the third is but little acted upon. No doubt the warm reception they would meet with in the stomach would effect their dissolution in a much shorter time, but probably still in the same sequence.

No. 10C is a form I have repeatedly made. The camphor should not have any oil or spirit to promote its division, as they make the mass both soft and crumbly. The camphor and extract of henbane, if vigorously worked together, make a satisfactory mass without any addition.

When it is desired to powder camphor without the addition of any fluid, a little powdered soap is a useful addition.

Camphor pills, and those depending for their efficacy upon creosote or essential oils, should be dispensed in bottles. We have here two samples of No. 10, both twelve months old; those kept in a bottle retain their original odour and activity, while those in the box have shrivelled away, and there is little left but the soap and the powder in which they were rolled.

## No. 11.

R.	Extr. Coloc. Co.	.	.	.	.	℥ij.
	Pil. Rhei. Co.	.	.	.	.	ʒj.
	Pulv. Ipecac.	.	.	.	.	gr. x.
	Extr. Anthem.	.	.	.	.	℥ij.
	Ol. Anthem.	.	.	.	.	gtt. x.

M. ft. Massa. in Pil. xl. div.

Exemplum 19,781.



The oil being rubbed with 5 grains of powdered soap, and then the colocynth, rhubarb, and ipecacuanha added, formed a good mass, which became crumbly immediately upon adding the extract of chamomile. The addition of four drops of liquor of potash perfectly restored its plastic condition.

A second box of the same was made as follows:—

The colocynth, rhubarb, ipecacuanha, and extract of chamomile were rubbed together, and then the oil, previously mixed with 5 grains of powdered soap. The mass was in good condition till the oil and soap were added, it then became very crumbly, which defect was in great measure remedied by adding 4 drops of spirit.

A third box of pills, made by the same prescription as last, but with almond meal instead of liquor of potash, overcame the crumbly nature of the mass.

The potass in the former case, and the emulsin of the almond in the latter, acting the same part in the pill mass which they do when used in the production of emulsions, *i.e.*, effecting the intimate and permanent mixture of oily and aqueous materials. Regard must be had to the tendency which almond meal has to impart hardness and insolubility to the mass if used in any large proportion. Liquor of potash, though a powerful chemical, is not so objectionable as might at first sight appear, for in the quantity used it probably does little more than neutralise any acidity in the extracts.

#### No. 12.

##### Dinner Pills.

R	Pulv. Rhei	.	.	.	.	℞j.
	Pulv. Myrrhæ	.	.	.	.	gr. xv.
	Extr. Anthem.	.	.	.	.	āā
	— Aloes Aquosi	.	.	.	.	℞ss.
	Ol. Anthemidis	.	.	.	.	gtt. xvj.

M. et distribue in pilulas xx.

Exemplum.

The oil works out of the pills unless its retention is effected by the addition of liquor of potash, almond powder, or yellow wax.

A small quantity of wax used in formulæ of this kind readily emulsifies with the other ingredients of the pill, especially if soap also be present. The quantity used should be so proportioned as to impart a soft gelatinous condition to the oil before the other ingredients are added.

## No. 13.

R	Ext. Colchic. Acet.	.	.	.	} $\begin{matrix} \text{āā} \\ \text{gr. xviii.} \end{matrix}$
	— Coloc. Co.	.	.	.	
	— Hyoc.	.	.	.	

ft. Pil. xij.

These ingredients, when mixed, are too soft for convenience. They have been dried to give them a suitable firmness, and the pills kept in an ordinary pill-box.

No. 14 are the same in every respect, but kept in a bottle.

No. 15 are the same, but with the addition of a small quantity of powdered tragacanth to give the desirable firmness.

No. 13 became soft by re-absorption of moisture, and fell out of shape; now by long keeping they are hard, but misshapen. Nos. 14 and 15 have both retained their shape tolerably well, and are hard.

Immersed in cold water, they all speedily show signs of dissolution; No. 14 a little more, and No. 15 a little less rapidly than No. 13.

## No. 17.

R	Extr. Taraxaci	.	.	.	gr. iij.
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ft. Pil. Mitte xxiv.

Exemplum.

Reduced to a suitable consistence by drying, and preserved in a box.

No. 18, the same as above, but with the addition of  $\frac{1}{2}$  grain of powdered tragacanth to each pill to give it sufficient firmness.

No. 17 speedily re-absorbed moisture and adhered into a lump, and is now a soft extractive mass without any appearance of ever having been made into pills.

No. 18 have retained their shape satisfactorily, and are now rather hard, but are readily acted upon by water.

Tragacanth, if in too large proportion, gives the mass a retractile character, and the pills, after being rolled round, draw back into short cylinders, and are with difficulty made to retain the spherical form; they also become very dry, hard, and less soluble than we could wish; but these objections only apply to cases where it has been used with want of judgment; if used in moderate quantity, and in conjunction with hygroscopic or readily soluble materials, it is a most valuable excipient.

The condition of No. 17 should be a caution to you not to trust to drying hygroscopic extracts to a so-called "suitable consistence." The consistence which is really the most suitable for them to possess, is such as gives them the least disposition to change their consistence by ordinary keeping. If they are then too soft, the requisite firmness is best given by their union with firmer drugs. You will remember in this connection the experiments I showed you in my lecture on drying.

The next four prescriptions have been selected to illustrate cases in which the materials are too hygroscopic to be safely trusted without the addition of some such excipient as powdered tragacanth.

R	Extr. Colchic. Acet.	.	.	.	} āā gr. j. gr. ij.
	Hyoscyami	.	.	.	
	Pil. Hydrarg.	.	.	.	
M.	ft. Pil. (c. G. Tragac. gr j.)				
R	Extr. Colchic. Acet.	.	.	.	} āā gr. iv. gr. ss.
	Pil. Hydrarg.	.	.	.	
	P. Ipecac.	.	.	.	
ft. Pil. ij.					
R	Extr. Colchic. Acet.	.	.	.	} gr. ss. āā gr. vj.
	Conii	.	.	.	
	Hyoscyami	.	.	.	
ft. Pil. iv.					

R	Pil. Hydrarg.	.	.	.	.	℥j.
	Pulv. Ipecac.	.	.	.	.	gr. vj.
	Ext. Colchic Ac.	.	.	.	.	} āā
	Hyoscyami	.	.	.	.	
ft. Pil. xij.						

## No. 19.

R	Ol. Lastreæ Filicis Maris	.	.	.	.	gtt. xvj.
	Sapo. Venet	.	.	.	.	gr. viij.
P. Amyli q. s. ut fiat. pilul. No. x. Sumat pilul mane.						

In this instance well-judged excipients have been selected by the physician; the soap combines well with the liquid extract of male fern, and gives it a ready emulsibility, while the requisite firmness is imparted by the starch.

## No. 20.

R	Bals. Copaib.	.	.	.	.	℥vj.
	Pulv. Cubebæ	.	.	.	.	℥j.
	Ferri. Carb.	.	.	.	.	℥j.
ft. Electuarius.						

Sign. A teaspoonful night and morning.

Was altered by desire of the patient to the following :—

R	Bals. Copaibæ	.	.	.	.	℥vj.
	P. Cubebæ	.	.	.	.	℥j.
	Ferri Carb.	.	.	.	.	℥j.
	Cera Flav.	.	.	.	.	℥jss.
	Magnes. Calc.	.	.	.	.	℥jss.
M. Ft. pil. ccxvj. Sign. Six for a dose twice or thrice a-day.						

Melt the wax, add the balsam, then the magnesia, and lastly the cubebs and iron.

Wax and magnesia are the orthodox excipients for making balsam of copaiba into pills. It may be the best known method of effecting that end, but if so bad is the best.

I examined the solubility of these pills after they had become firm, and found they would bear a week's immersion in cold water without appreciable change, and even boiling in water and in strong hydrochloric acid had little effect upon them. But when about fifteen months old they disintegrated by a week's immersion in cold water; they were not

much affected by boiling for a few minutes in water, but dissolved with effervescence in boiling hydrochloric acid.

The magnesia having become carbonated by long keeping, no doubt facilitated the disintegration of the pill. In the action of the acid this may perhaps be accounted for by the effervescence acting mechanically in tearing the pill open. Perhaps it would be better to use carbonate of magnesia instead of calcined in such formulæ.

		No. 21.	
R	Ferri Sulph.	.	gr. j.
	Pil. Rhei. Co.	.	gr. vij. M.
ft.	Pil. ij.	Mitte xvj.	
		22,183.	

The addition of sulphate of iron to a mass containing soap gives it a crumbly nature, which is very inconvenient in rolling out.

The addition of caustic of potash would remedy the physical defect, but at the sacrifice of chemical integrity, and our problem is to find some material which facilitates emulsification, or the union of oily and watery materials, without decomposing sulphate of iron; almond meal is the substance which naturally suggests itself, and with satisfactory results. The addition of four grains of almond meal and eight drops of water reduces this refractory mass to a workable condition.

As a general rule, masses which threaten trouble from their crumbly nature, may be worked if made a little softer than usual; and this expedient we are the more willing to adopt as the crumbly condition and the tendency to fall out of shape do not commonly occur together.

Many soluble crystalloids are difficult to render plastic with the usual aqueous excipients, but may be made into very good condition by means of some excipients of a resinous nature. Unguentum resinæ may be conveniently used for sulphate of iron, iodide of potassium, &c.; and though it cannot claim to be an elegant excipient, it is not more objectionable than castor oil, which is official in Plummer's pill.

The insolubility in water may be raised as an objection to excipients of this class, but if they are no less fusible than resin ointment there need be little fear of the stomach being incompetent to dispose of them as it does of other fatty matters.

## No. 22.

R.	Argent. Oxydi . . . . .	gr. iij.
	Extr. Belladon. . . . .	gr. $\frac{1}{4}$ .
	Ext. Hæmatoxyli . . . . .	gr. iss.
M.	ft. Pil. quotidie ante prand. sumend.	

Oxide of silver, and red oxide of iron are apt to give trouble, especially in combination with astringent materials, such as extract of logwood or extract of hops. If the mass is made very soft and worked off rapidly, it may be divided without difficulty ; but the pills may thus be inconveniently large, and become very hard by keeping. Castor oil and powdered soap worked into a soft pulp makes a convenient excipient for such materials ; the mass thus made works easily, the pills retain their shape well, and are plastic and freely soluble at the end of twelve months.

## No. 23.

R	Acid Carbol.	.	.	.	gr. xxiv.
	ft. Pil.	xxiv.			
	Aq.	.	.	.	gtt. j.
	P. Saponis				
	Cera flav. in lamina,	.	.	.	ā gr. xv.
	P. Rad. Taraxaci	.	.	.	gr. xxiv.

The acid rubbed with the water became liquid; the soap and wax converted this into a soft emulsible paste, to which a suitable firmness was imparted by the use of the taraxacum powder. The spongy nature of taraxacum, and its freedom from active properties, make it very suitable as an absorbing material for soft masses.

## No. 24.

R. Acid Carbol. . . . . gr. iij.  
ft. Pil. i. . . . . Mitte xlviij.

The carbolic acid having been increased to 3 grains in a pill presented more difficulty, but the task was accomplished in the following way.

144 grains of purest carbolic acid, and 24 grains of yellow wax, were melted together in a test tube, poured into a cold mortar, and rubbed into a rather adhesive powder; soft soap was then added by small degrees till 8 grains had been added, when it was found that the whole could be worked into a mass and divided into pills.

The pill mass having been formed by suitable excipients, is next to be rolled and cut. Some masses, such as contain watery extract and essential oil, or soluble crystalloids, are very apt to crumble under the roller. When this is the case they may be better rolled under the hand, the yielding nature of the skin helping to keep the mass together; they will even then probably crumble in cutting, and if this be anticipated, it is better not to roll them under the cutter, but simply to divide them with pressure between the two cutting surfaces, and round them by rolling between the fingers and thumbs. A dexterous operator will roll four at a time, taking two between the fingers and thumb of each hand, and will keep the others in a plastic condition while he is doing it by holding them in the palms of his hands with the two outer fingers.

Masses which are in good condition may be rolled into pills almost perfectly round and smooth if the machine be selected of a suitable size. Imperfectly formed pills may be finished by rolling them under a pill finisher, which is a circular piece of wood with a rim, similar in shape to the lid of a turned wood box. The pills being placed upon a slab and covered with the finisher are to be rolled with a light pressure in what may be called a double circular direction, which we may compare to the path of the moon moving in small circles round the earth, and in large ones round the sun at the same time.

It is not generally desirable to send out pills without some description of covering; liquorice powder or magnesia used to be the materials chiefly employed for this purpose, but are objec-



tionable on account of their stickiness ; too much adheres to the pill, and is unpleasant in the mouth. *Lycopodium* covers the pill most perfectly, and at the same time forms no clots, but has a rather unpleasant roughness in the mouth. The large grained starches, such as potato flour and *tous-les-mois* and powdered French chalk, are the materials I prefer for general use. Sometimes the nature or condition of a pill will determine the coating powder, as when a pill is softer than desirable, rolling it in powdered *tragacanth* removes the stickiness and gives it an exterior firmness.

The most elegant coating is silver or gold leaf, the use of which is an old custom, which has been recently revived. To silver a dozen pills put a leaf of silver into a covered earthen pot ; put the pills upon a piece of paper, with a drop or two of mucilage, and roll them under the fingers till they become so sticky that they will not roll side by side upon the paper, but roll over one another, and would, if the rolling were continued, stick into a mass ; when in this condition, throw them into the pot upon the silver, put on the cover, and shake violently. They will then be found covered pretty perfectly with the leaf, and any loose particles may be blown away.

If these instructions be not exactly followed, the coating is likely to be imperfect ; and if once imperfectly coated, it is almost impossible to make any better of it without commencing *de novo*, using a very stiff mucilage, or mucilage and powdered gum alternately, till the pills are *very* sticky, and then shaking them with a fresh leaf of silver.

Various other methods of coating pills have been introduced within a few years. I can only allude to one, which I can say from constant use answers perfectly and without any trouble or difficulty.

A tincture of sandarach is made by dissolving 1 oz. of the gum in 3 oz. of ether. A few drops of this are put into a covered pot, the pills thrown in, instantly shaken, and turned out upon a tray to dry for a minute, and are then ready for the box.



Pills coated in this way may be kept in the mouth for a minute without being tasted, but the coating yields to the action of moisture in a few minutes, and the coating with silver leaf yields even more readily ; there is consequently no fear of a pill losing activity from either of these coatings, provided the solubility of the pill itself is satisfactory.

### *Powders.*

The dispensing of powders is a very simple operation, and requires but little comment.

If a single powder of one ingredient is ordered, there is nothing to be done but to weigh it, fold it in paper, and write the appropriate directions.

If it contain two ingredients, for appearance' sake they should be mixed to look uniform, a rubbing with the knife upon the paper being sufficient for that purpose.

If there are several powders of mixed composition, the ingredients should be rubbed together in a mortar ; a thorough union is much more readily obtained by its use, and it is not a matter of indifference in what order the ingredients are put into the mortar. Thus, in the following :—

R	Calomel	.	.	.	.	gr. xij.
	P. Sacchari	.	.	.	.	ʒj.
	ft. Pulv.	xij.				

Do not put them in in the order in which they are written. Put the sugar in first and the calomel upon the top of it ; the mixture will sooner be uniform, and less calomel will adhere to the mortar.

R	Bismuth Tris. Nitr.	.	.	.	ʒij.
	Magnes. Carb.	.	.	.	ʒj.
	P. Sacchar.	.	.	.	ʒj.
	Ol. Caryophyli	.	.	.	℥xij.
M.	Ft. pulv.	xij.			

Put the sugar in first, drop the oil upon it, and rub them together ; then add the magnesia, and lastly the bismuth,

mixing between each addition. As a general rule put the lightest, roughest, bulkiest, or least active constituent in first, and materials of the reverse qualities in last. Excepting where some powerful ingredient is ordered in small quantity, and it is desirable to insure its thorough division and uniform distribution by rubbing it with a portion of the less active matter first, *e.g.*, a powder similar to the last with the addition of 3 gr. of opium, the opium would be rubbed with the sugar till smooth and uniform, then the oil, then magnesia, and the bismuth as before.

When the ingredients are mixed it is customary with some dispensers to guess out the quantity for each upon the papers arranged on the counter, and trust to the accuracy of the eye to detect a powder which is larger or smaller than the rest. Though some people can acquire a great degree of accuracy and dexterity in thus dividing the doses, it is not desirable to trust to it. The habit of weighing is most commendable, not only because of its insuring accuracy in the division, but, if a patient fancies that the powders act more at one time than another, it is a great satisfaction to both dispenser and patient to know that each one was accurately weighed; and the weighing out of twelve powders of ℥j. each, when the whole bulk weighs ℥iv., is a good practical lesson in accuracy and a useful check upon the correctness of the first weighing—the omission of an ingredient or the doubling of a quantity is thus guarded against.

If a powder is prescribed in bulk it should be sent out in a bottle, unless there be instructions to the contrary.

Volatile powders, such as carbonate of ammonia, sometimes prescribed in effervescing draughts, &c., should be enclosed in a wide-mouthed bottle, whether they are sent out divided or bulk.

Deliquescent powders may be dispensed in the same way, but it is desirable in this latter case to use the additional precaution of folding them in waxed papers.

## EXAMINATION QUESTIONS.

What is the object of making medicine into pills ?

What is the characteristic difference between the mucilages of tragacanth and acacia as excipients ?

In what cases should tragacanth be used, and in what quantity ?

What are the mechanical elements of a good pill mass ?

In what respects do sugar and glycerine compare and contrast as excipients ?

What objection is there to the use of pure glycerine as an excipient ?

What excipients are suitable for making oily materials into pills, either alone or in combination with watery extracts ?

What consistence is the most suitable for any given extract ?

What objection is there to reducing hygroscopic pills to a suitable consistence by drying ?

What species of excipient is most convenient for giving a suitable consistence to crystalloids soluble in water ?

## RECAPITULATION.

The object of forming a drug into pills is to have it in convenient doses, easily taken and not liable to change by keeping.

The physical qualities of a good pill mass are such as permit of its being readily formed into shape without crumbling, or subsequently falling out of shape or losing solubility.

These qualities are usually best attained by the presence of a fibrous material, a mucilaginous element, and water.

Gum acacia is suitable for giving adhesiveness when a fibrous material is present. Gum tragacanth gives retentiveness of shape where fibre is absent, and is especially suited to give firmness to hygroscopic extracts. It is best used in quantities no larger than absolutely required.

Glycerine and sugar tend to preserve the solubility of a

pill ; the former retains its softness also, and if not sparingly used, is objectionable on account of its hygroscopic nature.

Essential oils and analogous bodies require wax, soap, or other excipients of an emulsifactive nature, to enable their free mixture with aqueous pill masses.

Hygroscopic materials, when ordered in pills, should be protected against deliquescence by the addition of tragacanth.

Crystalloids soluble in water, and some other materials not easily worked with aqueous excipients, are more tractable in combination with oily and resinous.

## LECTURE XXIII.

### OINTMENTS, PLASTERS, SUPPOSITORIES, &c.

Dispensing Ointments, Plasters, Suppositories, &c.—Ointments, Gritty Materials, Extracts, Watery Materials—Plasters, Spreading, Shapes, Adhesive Margins, Blisters, Prescriptions—Suppositories, &c., Cacao Butter, Lubricants for Mould, Tin Foil Moulds.

THE dispensing of ointments is one of the very simple operations of pharmacy: it rarely involves the melting of the ingredients, usually only the mixing of two or more materials together, one of them at least being unctuous, and the chief aim is to ensure perfect smoothness and uniformity. Dry materials should be reduced to the finest possible powder, if not dissolved before being added to the grease. Watery extracts, where such are prescribed, should be reduced with water, glycerine, or spirit, to a consistence at least as soft as the grease with which they are to unite.\* Chemicals should be protected against the chance of decomposition, which might in some cases take place from contact with steel knives or marble slabs or mortars.

Ointments are best sent out in covered earthen pots; wooden boxes are only fit for firm ointments, and even in that case soon become offensive. Soft ointments are best covered with waxed paper before putting the lid on the pot, to prevent the lid becoming soiled in carriage; but if the ointment is firm, the use of paper is better avoided, as it becomes disagreeable itself if left there, and answers no useful purpose.

\* Ointments made with extracts reduced with water frequently turn mouldy with keeping. It is not unlikely that the use of spirit or glycerine for softening the extract might overcome this objectionable feature.

I will illustrate with a few prescriptions selected from actual practice.

R	Hydrarg. precip. rub.	.	.	.	3iij.
	Cerat. cetacei	.	.	.	3j.
	Ft. ung.				
R	P. boracis	.	.	.	3ij.
	Ung. cetacei	.	.	.	3ij.
	Ft. ung.				
R	Potassii iodid.	.	.	.	9ij.
	Adipis	.	.	.	3 ij.
M.					

In the first of these the powder must be rubbed as fine as possible in the dry state; \* it may then be transferred to a slab to be mixed with the ointment, or the ointment may be added in the mortar. It is scarcely possible to make the powder as smooth by rubbing on a slab with the grease, but they may be made smooth by grinding with a small portion of the grease in the mortar, though in some cases this is scarcely practicable. The borax may be obtained fine by being effloresced, and the red oxide of mercury by being precipitated in the wet way; but these are scarcely legitimate deviations from the customary practice, unless the sanction of the prescriber can be obtained. Borax may also be made into a smooth ointment by dissolving it in glycerine.

In the third example the iodide is equally difficult to rub smooth, unless with the addition of a little water, which may be considered admissible, as it is adopted in the official ointment of iodide of potassium.

R	Ung. hydrarg.	.	.	.	.	} āā } 3ss.
	Extr. belladon.	.	.	.	.	
M.	Ft. ung.					

\* Unless kept ready for use by moist grinding and subsequent drying, as described in my lecture on grinding.

R	Extr. belladon.	.	.	.	.	℥jss.
	Acid. hydrocyan. Sch.	.	.	.	.	℥ss.
	Adipis	.	.	.	.	℥j.

M. Ft. ung.

The extract of belladonna of commerce is usually soft, but if it be of anything approaching a good pilular consistence, it must be diluted with water till at least as soft as lard; it will then pretty readily mix with a knife and slab. In the second ointment the extract and lard should be mixed first, and the acid added by drops while the ointment is being rubbed on the slab, so as not to allow the acid to be long exposed to the action of the air. When once thoroughly mixed with the grease, it is much protected from evaporation. Probably an alkaline cyanide, instead of hydrocyanic acid, would be a change for the better.

R	Liq. plumbi.	.	.	.	.	} āā
	Glycerin	.	.	.	.	
	Ceræ. alb.	.	.	.	.	℥iv.
	Atropin.	.	.	.	.	gr. iv.

M. Ft. ung.

In this case, it being found quite impracticable to mix white wax into an ointment with these ingredients, it was supposed that ceræ. had been intended for ceratum, which was consequently used, and a satisfactory ointment produced, though even then there was a little separation after keeping. This was entirely obviated by using an equivalent of the diacetate of lead in powder instead of the ℥ij. of liquor.

It may just be noted in passing, that the addition of mucilage, which gelatinises with the solution of diacetate of lead, enables its combination with ointment in any proportion.

No doubt almond powder might have been satisfactorily used in this case as in the following.

R	Zinci. oxydi.	.	.	.	.	℥ss.
	Liq. atropiæ	.	.	.	.	} āā
	Liq. morphiæ hydrochl.	.	.	.	.	
	Ol. olivæ	.	.	.	.	℥ij.

M. Ft. embrocatio ter die utend.

The oil and oxide being mixed together, the watery liquids refused to unite with them.

The watery liquids being mixed with the oxide, and the oil stirred in, at first all mixed, but the liquors afterwards separated again.

It thus became necessary to add some excipient capable of emulsifying the whole. Soap would, no doubt, have effected this object, but with the decomposition of the hydrochlorate of morphia, as this was not admissible, we had recourse to the following method with perfectly satisfactory results :—

Take a Jordan almond, and having scraped the brown skin off, reduce it to powder and rub it with the oxide of zinc and the liquors till quite smooth, then add the oil, rubbing with a knife upon an ointment slab, till the whole forms a smooth and uniform ointment.

Almond powder (free from the skin) would no doubt be equally efficacious in mixing other aqueous fluids with oil or ointments.

### *Plasters.*

The spreading of plasters is an art of which little can be learned by verbal instruction. Practical demonstration on the part of the teacher, and practical experience on the part of the learner, are the only means by which the art of spreading plasters with facility can be acquired.

I will first draw your attention to a few points, and then proceed to give practical demonstration.

Plasters are spread upon paper, textile fabrics, or leather; in dispensing, the latter is almost exclusively used. The mode of spreading surgeons' strapping, ordinary adhesive plaster on calico, is not of sufficient importance to the retail pharmacist to warrant our spending time over it, we will therefore confine our attention to the spreading on leather. The leather used is known in commerce as sheepskin, split skin, lamb skin, and kid skin; the first being used on all ordinary occasions, the others being used where thin and soft plasters are



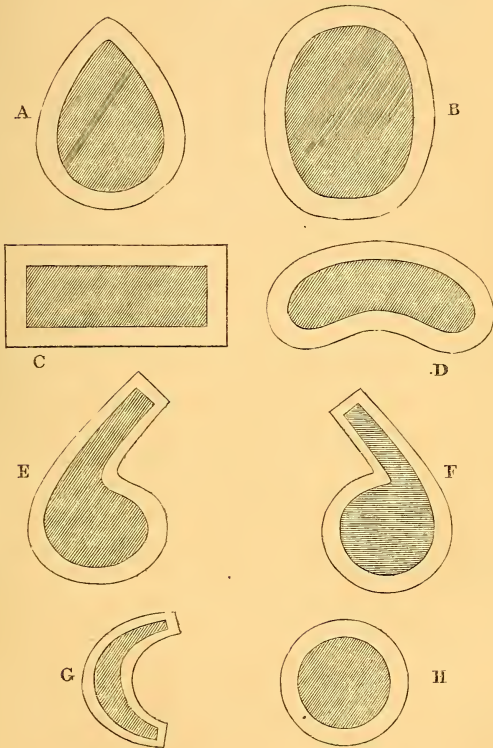
specially required. In selecting a piece of leather, it should be taken from a part of the skin which will enable it to be cut of pretty uniform thickness and *flat*. The edges of the skin are usually stretched, and though they may be used with a little extra care and skill, they are apt to draw back more or less to the shape they originally possessed upon the living animal. Split skin is frequently used where a plaster is required as a protection to a bed sore, and for this purpose the plaster is usually spread close to the edge of the leather, as a loose margin is liable to become folded under the patient, much to his annoyance; this necessitates the spreading of the plaster as thin as possible to avoid its melting with the heat of the body, and running out beyond the leather upon the clothes.

The leather having been selected and cut, is to be laid upon a quire of brown paper, which forms a bed of a convenient degree of softness, and which does not abstract the heat of the plaster too rapidly. The leather should then be smoothed with a gently heated spatula, the requisite degree of heat being judged by its just sufficing to throw off a drop of water in the spheroidal state, or by the sensation of warmth it produces when held near the face. The inexperienced operator will judge best by the effect the spatula has upon a fragment of leather; if the heat discolours it, or causes it to curl up, it is too hot; if it does not smooth the leather, the heat is insufficient. In using an ordinary plaster iron, the size should be proportionate to the size of the plaster; a large iron is clumsy for spreading a small plaster, especially if it is to be done without a paper shape to cover the margin; and a small iron will not retain heat enough to spread a large plaster. Spatulas are also made of brass, hollow, and supplied with gas by which the heat can be maintained any length of time. By this arrangement a moderate sized instrument may be used for a plaster of any dimensions, or for any number of plasters in succession without becoming cold. To set against this, there is the inconvenience of having an india-rubber tube

for the supply of gas always attached to the handle. Taken altogether, I find the simple iron generally preferred.

The shapes of plasters are determined by the part of the body to which they have to be applied, the following being the usual :—

Fig. 35.



A, for the chest, heart-shape. B, for the shoulders, oval. C, for the small of the back, oblong. D, side, kidney shape. H, knee, round. Ears, retort shape or crescent. F, right. E, left. G, either.

The plaster is melted by rubbing it against the hot spatula held upright with the tip resting upon a piece of paper; when sufficient is melted, it is pushed off the paper upon the leather which is to receive it, and the spreading effected without loss of time, by pushing the melted plaster over the leather, the latter being turned about that the melted material may be pushed *from* the operator. The position of the spatula should be such that it presses upon the leather by the edge nearest the operator, and the pressure should be lighter in proportion as the plaster is more fluid.

Patterns of stiff paper or card may be kept to aid in drawing any of these shapes upon the leather previous to cutting.

In the spreading of plasters, neatness is much facilitated by the use of what is technically called a plaster-shape, or mould of paper, which consists of a sheet of paper with a piece cut out of the centre the size and shape which the plaster is required to possess.

Fig. 36.



All the forms given above, except those for the ears, are symmetrical, and may be cut equal-sided by doubling a sheet of paper, and making the fold represent the symmetrical axis, and then cutting both sides at once along the line that represents the edge of the plaster. For the heart shape, the sheet is cut as represented in fig. 36, and for the kidney shape, as in fig. 37. For the square, oblong, round, &c., the sheet may be folded twice, and the shape cut out of the centre of the paper, which constitutes then the folded corner. The round is cut as in fig. 38, the square as in fig. 39.

Fig. 37.



Fig. 38.



For spreading a plaster of a particular size and shape, the leather should be roughly cut somewhat larger than its ultimate size, to allow for the contraction which always takes place during the smoothing of the leather, and to allow also for trimming the edges when the plaster is finished.

Having cut and smoothed the leather, lay upon it the piece of paper cut from out of the marginal shape in exactly the place which is to be covered with the adhesive material, then, having dipped the marginal part in water, lay it carefully round the centre piece, without disturbing the position of the latter, then apply a little pressure, and the moist paper will adhere to the leather. The centre

Fig. 39.



piece being removed, the plaster is to be spread in the manner before described, and, as the operator's attention is not needed to keep it to the required shape, the process is easily and quickly performed, his whole attention being given to making it smooth and of uniform thickness. As soon as this is done, the paper margin is to be lifted at one edge and pulled off while the plaster is still warm. The trimming of the edge of the leather then completes the operation.

It is desirable that every one who has plasters to spread should also practise spreading them without the aid of the paper shape, as there are times when the ability to do so will be useful, such as the spreading of a belladonna plaster on leather with an adhesive margin, which is to be done in the following way:—First, cut the paper shape with the centre piece the size the belladonna has to extend, and fix this piece on the leather with water, as before described, then cut from the inner edge of the marginal piece a strip of the width it is intended to make the adhesive margin, and attach the marginal paper to the leather in the same way, leaving a uniform space between the two papers for the reception of the adhesive plaster. Then the resin plaster is to be spread upon this space, no matter whether or not it spreads also over part of the papers. The centre paper being then taken off, the belladonna plaster is to be spread in the space it occupied, taking care to bring it pretty close up to, but not upon, the adhesive margin.

Sometimes plasters requiring an adhesive margin are simply spread upon a piece of adhesive plaster on calico. This is the usual custom with blisters, the paper shape being

used as before, but the plaster instead of being melted and spread with the spatula, is spread with the thumb, no more heat being used than it gets from the hand. In cold weather cantharides plaster becomes crumbly, but its plasticity may be restored by working it in the hand for a minute, before using the thumb as a spatula, or by beating it with a gently warmed iron. When the spreading is finished, the surface may be smoothed with a warm knife, and a little dry cantharides powder rubbed in before removing the paper margin.

When compound plasters are prescribed the ingredients may be melted together in a small dish or galli-pot, in the same manner as in making the official compounds, or they may be simply melted by the aid of the plaster spatula upon a piece of paper or tin plate.

The following are a few examples of prescriptions for plasters:—

Plaster No. 1.

R	Empl. bellad.	.	.	.	.	℥iv.
	Picis	.	.	.	.	℥iv.
	P. camphoræ	.	.	.	.	℥j.

Misce fiat emplastr super alutam cum margine adhesivum magnetu  
iv polices. ☐

(One-fourth the quantity sufficed.)

No. 2.

R	Extr. belladon.	.	.	.	gr. xij.
	Mellis	.	.	.	℥ss.
	Empl. belladon.	.	.	.	℥iiss.

M. Extende super alutam.

No. 1. The belladonna and pitch plasters being melted upon a tin plate, the camphor was stirred in with as little heat as possible.

One-fourth of the quantity of material was sufficient. It may be stated, as a general rule, that one drachm of material is sufficient for ten square inches of surface.

No. 2. The extract and honey were mixed upon a tin plate by means of the spatula, and then the plaster added and all

mixed together. This union of watery and resinous constituents is not desirable, as it destroys the adhesiveness.

The same objection also applies to the next, fig. 40.

Fig. 40.



In fig. 41 (p. 306), the point requiring attention is to have the tartar emetic in the finest possible state of division, and thoroughly mixed with the pitch plaster before it is spread.

*Suppositories, Medicated Pessaries, and Bougies*

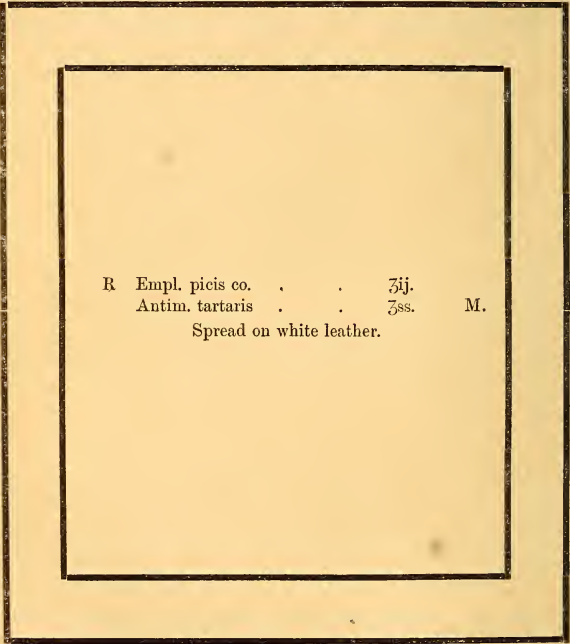
Are somewhat troublesome articles to be produced extemporaneously. The two latter are recent additions to pharmacy; the former are of old date, but much more frequently used recently.

Every one ought to be able to turn out a passible suppository of any required size, shape, and composition, at short notice.

The active matters were formerly combined with gum, &c., in the same manner as pills are made; but as they often contained matters which were adhesive, and not absorbed by the

bowel, they sometimes left a residue which was objectionable, as not being readily removed. Cacao butter has now almost entirely superseded these mucilaginous excipients, though it has recently been proposed to substitute a compound of gelatine and glycerine in some cases. By desire of one of our local medical men, I dispensed a number of gelatinous suppositories a short time ago ; but as they appear now to be discontinued, I presume they were not found to offer any material advantage.

Fig. 41.



R   Empl. picis co.   .   .   ʒij.  
      Antim. tartaris   .   .   ʒss.   M.  
                    Spread on white leather.

I will leave out of the question for the present all excipients except cacao butter, with the small additions of wax, oil, lard, or spermaceti, which are usually made to it.



Powdered materials, such as opium, oxide of zinc, or tannin, should be rubbed smooth with a small quantity of oil or lard upon an ointment slab, while the cacao butter is being melted, with the addition of about  $\frac{1}{4}$ th of its weight of wax or spermaceti, in a small water bath. When the grease is melted, it should be allowed to cool again, so far as to become slightly opaque before the other materials are added; they may then be stirred in with a teaspoon, and when mixed, and so far cool as to be of the consistence of thin cream, may be poured into the moulds by teaspoonfuls, stirring between each to insure the uniform distribution of the active matters.

Many dispensers have experienced difficulty in detaching the suppositories from the mould, as they are apt to adhere, and are broken in the attempt to extract them. They are usually cast in metal moulds made in two pieces, the conical hollows occupying the junction, so that, when the pieces are separated, the extraction of the suppositories is facilitated, and may usually be effected without much difficulty if the mould be well polished and well shaped; if it be very cold when the melted grease is poured in, and allowed to become quite cold again before it is separated; and if the material be not too warm when poured in, and have a sufficiently firm consistence when cold. As some of these conditions depend upon the season of the year, &c., the dispenser cannot always command the means of certain success without some little variation in the mode of working. Many suggestions have been made to obviate this difficulty; the simplest and most obvious is to rub the mould over with soap and water, or glycerine (not oil) or water. To prevent a watery extract adhering you would naturally use oil; but to prevent a greasy material adhering, watery lubricants are best. Water itself would answer perfectly if it could be made to cover the surface of the mould without running off in drops; and this may in some measure be accomplished, if the mould be clean and cold, by breathing upon it, blowing through an india-rubber tube into each hollow just before pouring in the grease; but



success is more certain with the use of a small portion of soap and water. Another method I have used is to make moulds of tinfoil, into which the suppositories may be cast, and as soon as solid they may be immersed in a stream of cold water till hard; the foil may then be unrolled without difficulty or any risk of the suppository being broken. This mode of working is the best, when the size or shape ordered by the physician does not correspond with the moulds in use.

I have made forms for these tinfoil moulds by casting lead into an ordinary suppository mould, or by softening the end of a stick of sealing-wax, and forcing it into a conical minim measure, by which means the requisite conical form is obtained. The foil should be cut of a convenient size and shape for rolling round this form as smoothly as possible. The moulds thus obtained should be set upright in any convenient stand—if a regular suppository mould is at hand, nothing answers better; but in its absence, a row of ounce phials will do quite well, putting one of the tinfoil cones into the mouth of each.

Pessaries and bougies, differing from suppositories only in form, may be made upon precisely similar principles.

Suppositories are usually made to weigh about 15 grains each, and of a rounded conical form, the outline being represented by two convex curves. Pessaries are of the same shape, but usually weigh two drachms each. Bougies are cylindrical, about  $2\frac{1}{2}$  inches long, 0·2 inch in diameter, and weigh about  $\frac{1}{2}$  a drachm.

## LECTURE XXIV.

### QUALITATIVE TESTS OF THE PHARMACOPŒIA.

THE system of testing adopted in the Pharmacopœia has three distinct objects in view—the first, to ascertain that the material under examination is really that which it is expected to be; the second, to prove the absence or the presence of impurities which the substance in question is liable to contain; and the third, to determine that the correct quantity of the most important constituent is present. The instructions generally are such that these objects are satisfactorily accomplished even by an operator who imperfectly understands what he is doing, provided he yields implicit obedience to the instructions given. But however carefully they may be drawn up and followed, no one could expect that his testing would be considered satisfactory either by his customer, his merchant, or the Board of Examiners, unless he could explain the reason for each operation, and the meaning of each indication. Fortunately the subject is full of interesting lessons in experimental chemistry; I can scarcely imagine any subject more interesting, both practically and theoretically, to the pharmaceutical student than a systematic testing of the various articles in daily use, according to the instructions of the Pharmacopœia, with the aid of a chemical handbook to explain the theory of the reactions in those cases where they are not at first sight self-evident. I will now proceed to lay before you such illustrations as will exhibit the diversity of the methods used for proving the freedom from impurity of pharmaceutical products.

I shall not attempt to go through all the materials to be tested, nor all the test solutions, nor yet all the tests which are

used in those cases which I select for illustration, but simply to pick and choose among the mass such as best serve my purpose, which is to sketch out the manner in which you should study the paragraphs on "Characters and Tests," appended to the chemicals in the Pharmacopœia, to illustrate the actual reaction in a few cases, and to throw a little light upon the unexplained object sought in a few others.\*

### *Acetum.*

The first chemical in the Pharmacopœia is vinegar, and it gives us a fair illustration of the diversity of methods of examination, and the many little points which should claim our attention in critically examining the common articles of pharmacy. After noticing its brown colour and characteristic odour, with which the pharmacist is expected to be familiar, we are informed that its specific gravity is 1·017 to 1·019. These densities do not indicate any precise strengths, because they are not purely the result of the acid it contains. It is sure to contain some fixed matter in solution, which will add to its density, and is very likely to contain some spirit, which will detract from it. The examination of its specific gravity is therefore a test to which it is not frequently subjected; its strength being much more accurately determined by its power of neutralising a given quantity of caustic, soda which is the test next brought under our notice. A fluid ounce of vinegar should require 402 grain-measures of the volumetric solution of soda for its neutralisation, in which case it will contain 4·6 per cent. of anhydrous acetic acid.

In testing vinegar with chloride of barium, a precipitate is usually formed from the presence of sulphuric acid; but as a small percentage of sulphuric acid is not objectionable, provision is made that a fluid ounce of vinegar, after having been mixed with ten minims of solution of chloride of barium,

\* My readers, while they have one eye upon these pages, should have the other on the Pharmacopœia, otherwise they will be taking an antidote without the poison, a thing not often desirable.

British Pharmacopœia (1 in 10), and filtered from any sulphate of baryta which may have precipitated, shall not give a further precipitate with more of the chloride, proving that all the  $\text{SO}_3$  in the ounce of vinegar had been precipitated by 1 gr. of chloride; consequently that there was not more than 0.4 gr. sulphuric anhydride in the ounce.

The absence of coloration, by the action of sulphuretted hydrogen, indicates its freedom from a considerable group of metals, copper and lead being the most likely metals of this group to be present. Iron, which is an equally probable impurity, is not indicated by sulphuretted hydrogen unless the acid be neutralised.

You will perceive that to go through the tests of the Pharmacopœia in this way would be much too lengthy a task for an evening's lecture; but it is what every pharmaceutical student should do in the regular course of his study. The limited number of reactions upon which it is practicable for me to make any comment necessitates my limiting my selection to a few of those only which either illustrate a widely applicable test, or are not sufficiently clear to insure their full comprehension by a student of ordinary intelligence.

*Acetic Acid* is directed to be quite free from sulphuric acid, as proved by its giving *no* precipitate with  $\text{BaCl}$ . And the absence of hydrochloric acid is also to be ascertained by its producing no precipitate with  $\text{AgNO}_3$ .

Though sulphuric acid and chlorine are not the only chemicals which are respectively precipitated by  $\text{BaCl}$  and  $\text{AgNO}_3$ , they are almost exclusively the substances sought for by these reagents in the testing enjoined in the Pharmacopœia.

It is, of course, useless to attempt any testing without first making sure that the impurities which are to be sought for in the material under examination are not introduced by the use of impure reagents, impure water, or imperfectly cleaned utensils. All ordinary water, except distilled water, and sometimes even distilled water itself, will give precipitates with  $\text{BaCl}$  or  $\text{AgNO}_3$ .

Acetic acid is also to be tested by mixing with pure hydrochloric acid and granulated zinc, and examining the evolved hydrogen for HS which it would contain if the acetic acid contained sulphurous acid.

Glacial acetic acid is required to solidify at 34° Fahr., and not to melt again under 48° Fahr. The commercial acids frequently have higher freezing and melting points, indicating consequently greater strength.

*Acid. Arseniosum.*

Arsenious acid, if pure, is entirely volatile. Complete volatilisation is used as a test in various other cases, such as the mercurial salts, ammoniacal salts, except phosphate (and some others which are not medicinal), also for iodine, benzoic acid, &c. But as entire volatilisation does not prove the absence of other volatile substances, the test is supplemented with others. In the case of arsenious acid, with a volumetrical examination with iodine; in the case of benzoic acid, with the examination of its melting (248° Fahr.) and boiling points (462° Fahr.)

*Acid. Citricum.*

Citric acid is examined for tartaric acid, its most probable impurity, by the addition of a solution of acetate of potass, which yields a precipitate of cream of tartar if any tartaric acid be present.

This method of detecting a suspected or probable impurity, by the addition of a substance with which it forms a precipitate, if present, is one of the most generally applicable modes of testing. It must not be overlooked that citric acid produces a precipitate of cream of tartar, if added to a solution of Rochelle salt or soluble tartrate of potass, as will sometimes occur in dispensing.

*Acidum Gallicum.*

This is examined for tannic acid, which, on the addition of

a solution of gelatine, produces a leathery precipitate with the latter acid, but none with the former.

*Acid. Hydrochloricum.*

It is essential to dilute it with water before using BaCl as a test for  $\text{SO}_3$ , the chloride of barium being precipitated by the pure acid in a concentrated state. Sulphuretted hydrogen is used as a test for various metals with which it is apt to be contaminated, more especially iron and arsenic, the former of which will give a black precipitate if the acid be neutralised with ammonia after the use of HS. Arsenic yields a yellow precipitate in a dilute acid solution, but not in an alkaline. The boiling a piece of bright copper with the dilute acid is a further test for arsenic, which, if present, is deposited as a steel-grey film upon the copper. Sulphurous acid is tested for in the same way as was pointed out in connection with acetic acid. It is an impurity which by other modes of testing is easily overlooked.

*Acid. Hydrocyanicum Dil.*

Is tested with nitrate of silver for HCl, the cyanide of silver which is thrown down being entirely soluble in nitric acid; any residue which may be left indicates the presence of HCl.

*Acid. Nitricum*

Is tested for sulphuric and hydrochloric acids by the use of BaCl and  $\text{AgNO}_3$ , and requires to be freely diluted to prevent the precipitation of nitrate of baryta, which is but sparingly soluble in strong nitric acid.

*Acid. Phosphoricum Dilutum*

Is tested for nitric acid, which might be present from careles making, by the addition of sulphate of iron, which forms a dark precipitate in the presence of a trace of nitric acid.

*Acid. Sulphurosum*

Is tested for sulphuric acid, which it always acquires by slow oxidation if long kept. Chloride of barium gives no precipitate unless sulphuric acid be present; but if chlorine be added also, the sulphurous is converted into *sulphuric* acid, and the precipitate is immediate and abundant.

I have found commercial sulphurous acid contain sulphate of lime as a crystalline deposit, the acid having no doubt been prepared by some other process than that of the British Pharmacopœia.

*Amylum.*

In relation to starch, I need only point out that the testing with cold water and iodine, which is intended to indicate granules injured by heat or otherwise, may give the blue colour by over-powerful friction, even if the starch be perfect in the first instance, as I pointed out in my lecture on Grinding.

*Bismuthi Carbonas.*

The presence of subnitrate is tested for with the sulphate of indigo, which is bleached by nitric acid, but not affected by pure carbonate of bismuth and pure sulphuric acid.

*Calci Chloridum.*

Chloride of calcium prepared by the Pharmacopœia process may contain either chlorinated lime or perchloride of iron. The former is detected by addition of HCl producing the odour of Cl; the latter by a brown precipitate being deposited on the addition of lime water.

*Calci Phosphas.*

The testing of phosphate of lime in solution in nitric acid with acetate of soda, is intended to detect the presence of phosphate of iron, which is equally soluble in the nitric acid; but when the nitric acid is converted into nitrate of soda, and acetic acid left free, the phosphate of lime remains dissolved in the acetic acid, while the phosphate of iron is precipitated.



*Ferri et Quinæ Citras.*

The white precipitate which takes place in a solution of this salt when ammonia is added is quinine, the oxide of iron remaining in solution; but when caustic soda is added, a brown precipitate takes place, because peroxide of iron is precipitated along with the quinine.

*Ferri Peroxidum Hydratum.*

The absence of a blue precipitate with red prussiate of potass indicates the absence of protoxide of iron as an impurity in the peroxide.

*Ferri Sulphas.*

Sulphate of iron, if entirely free from peroxide, gives a very pale blue precipitate with yellow prussiate of potass; the object of the test being to indicate by the paleness of the colour the comparative freedom from oxidation.

*Liq. Bismuthi et Ammoniac Citratis.*

Solution of potassa and hydrochloric acids both produce white precipitates in solution of citrate of bismuth and ammonia; the former being a hydrated oxide of bismuth, the latter is citrate of bismuth, which though soluble in citrate of ammonia, is not soluble in solution of chloride of ammonium or free citric acid.

*Potassæ Citras.*

The absence of an immediate precipitate when chloride of calcium is added to citrate of potass, indicates the absence of tartaric acid; but when the solution is boiled, precipitation takes place, because the citrate of lime is less soluble in hot water than in cold. The citrate of lime once deposited, is only slowly taken up again as the liquor cools; probably combination with water taking place at the same time.



*Potassii Bromidum*

Is tested with mucilage of starch and chlorine to prove the absence of iodine, which, though much the more costly element of the two, is sometimes present as an accidental impurity.

*Potassii Iodidum*

Is tested with mucilage of starch and tartaric acid, which will give a blue precipitate if iodate exist in the iodide, but not otherwise.

*Sodæ Bicarbonas.*

Bicarbonate of soda is tested with perchloride of platinum to prove the absence of potass and ammonia, impurities which it is not liable to contain under ordinary circumstances. With perchloride of mercury, a white precipitate indicates that the soda is saturated with carbonic acid, while a brown precipitate indicates that the proportion of carbonic acid to soda is less than two equivalents to one.

*Zinci Sulphas.*

Sulphate of zinc yields a white precipitate with ammonia, soluble in excess of the reagent ; and this reaction takes place equally well without the solution being first boiled with nitric acid, as directed in the Pharmacopœia, the object of this injunction being to insure the peroxidation of any iron present. The protoxide is not so perfectly precipitated by ammonia, and a small quantity might be overlooked, which would not escape detection if first peroxidised.

## LECTURE XXV.

### THE QUANTITATIVE TESTING OF THE PHARMACOPŒIA.

QUALITATIVE testing is designed to determine the presence of everything that ought to exist in the material under examination, and the absence of everything else.

Quantitative testing is designed to determine the quantity of a constituent known to be present in the substance under examination.

Quantitative examinations are either gravimetric, in which the quantity is estimated by weighing, or volumetric, in which the quantity is estimated by measuring the volume of a reagent required to produce a given change, the weight being afterwards ascertained by calculation.

In gravimetric analysis the constituent to be estimated is either separated and weighed in the separate state, or is brought into some new state of combination, the new compound being easily separated from the other constituents of the original material, and the quantity of the element sought is calculated from the proportion in which it is known to exist in the new compound formed.

We will consider one or two gravimetric examinations before going on to volumetric analysis.

#### *Ferri et Ammoniac Citras.*

The testing of this salt is one of the simplest possible cases of quantitative examination.

When exposed to a full red heat, till it ceases to change in weight, the residue should weigh 27 per cent. of the original

salt, the loss being water, ammonia, and citric acid, and the residue peroxide of iron. The only source of error in the operation being the chance of small portions being carried off in scintillations, or part of the oxide being reduced to the state of protoxide, by the action of the organic matter present; but, if the heat be continued, any protoxide at first formed again absorbs oxygen, and becomes peroxide. It is necessary to supplement this test, by ascertaining that the oxide of iron has not an alkaline reaction, which it would have if citrate of potass or soda were present in the original salt—organic salts of the fixed alkalis being converted into carbonate by incineration.

*Acid. Phosphor. Dil.*

To estimate the strength of this solution, 6 fluid drachms (the British Pharmacopœia says 355 grains, which is the weight of 6 fluid drachms) is poured upon 180 grains of oxide of lead in powder, the whole evaporated to dryness, and calcined. The oxide of lead fixes the phosphoric acid, but allows the water to evaporate, so that the weight of the two, after calcination, is the weight of the oxide of lead (180) plus the weight of anhydrous phosphoric acid present in the fl. ʒvj. of dilute liquor. For this test to be available, it is necessary to insure the absence of sulphuric acid, which is done by the qualitative examination which precedes. It is also necessary that the oxide of lead be in excess of the phosphoric acid. If there be more acid than the lead is capable of uniting with, some would be lost, as the free acid is in some degree volatile at high temperatures. The oxide of lead must be thoroughly dry before the first weighing, or the moisture which it contains will be driven off in the process of testing, and the ultimate weighing will indicate less than the true quantity of phosphoric acid.

*Ferrum Redactum.*

In the examination of reduced iron, the problem is to determine how much of the oxide of iron has escaped reduction.

This is accomplished by acting upon it with an excess of iodine, dissolved in water with the aid of iodide of potassium. The iron, which has been reduced to the metallic state, forms a soluble iodide, but the oxide remains insoluble, and, after being washed, dried, and ignited, should not weigh more than half as much as the original sample of reduced iron. It is quite practicable to obtain reduced iron with much less contaminating oxide than here allowed, but it must be remembered that one object of having iron in this fine state of division is to insure its ready oxidation, and, if it be reduced at a low temperature, it is impossible to keep it from absorbing oxygen, which may even be so rapid as to cause its ignition.

Reduced iron might also be tested by acting upon it with a given weight of pure iodine, so adjusted that there would remain a portion of iodine not combined with iron, if the sample under examination contained the required proportion of metallic iron.

*Liq. Antimonii Chloridi.*

Sulphate of antimony, treated with *strong* hydrochloric acid, evolves sulphuretted hydrogen, and becomes chloride of antimony; but in a feeble acid liquor, chloride of antimony, treated with sulphuretted hydrogen, precipitates sulphide of antimony, and hydrochloric acid is formed, the affinities of these elements being nearly balanced. To enable this precipitation to go on satisfactorily, the solution requires to be diluted, but the liquor of chloride of antimony is decomposed by dilution into an acid salt, which remains in solution, and a basic salt, which is precipitated; tartaric acid is added to the water with which the liquor is diluted, the tartrate of antimony formed, being a salt which is not decomposed by water, but which is still readily precipitated by sulphuretted hydrogen.

In all cases of precipitation, it is most important to take care that there has been sufficient of the reagent used to precipitate the whole of the element intended to be thrown down,

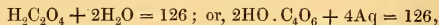
and equally necessary to wash the precipitate thoroughly, that its weight may not be added to by the presence of soluble salts which ought to have been washed out. With some precipitates the washing required is very considerable, and should be continued in any case till the water comes through so nearly pure as to give no precipitate with some delicate test for the bye-product.

*Potassæ Permanganas.*

Five grains of the permanganate of potass dissolved in water require for complete decoloration a solution of 44 grains of granulated sulphate of iron, acidulated with two fluid drachms of diluted sulphuric acid. Here we have a test which naturally brings us to the method of volumetric estimation, for we do not separate any element and weigh it, but simply estimate the quantity of one of the elements present by the quantity of a reagent which it is necessary to add to produce a certain visible change. The change in this case being the conversion of the highly-coloured permanganate into colourless sulphate of potass and sulphate of manganese, the oxygen of the permanganic acid converting the sulphate of iron from a pro- to a per-salt. A great variety of changes of this definite and visible character have been recently made available for determining the percentage value of pharmaceutical and commercial chemicals.

We will now turn to the apparatus and methods which have been adopted to facilitate the attainment of accurate results by volumetric analysis.

Pure oxalic acid consists of—



and forms salts neutral to litmus when the 2H is replaced by 2Na, or any other powerful base ; but as it is more convenient to work with single equivalents of the alkalis, we adopt 63—the half equivalent—as our working number. This weight in

grains or decigrams is to be dissolved in 100 measures of water, the measures being 10 grains or one gram each, according as English or French weights and measures are adopted. As the French weights and measures are very largely used now in analytical operations, I will use them only for the present.

It will be readily seen, that if we take an equivalent in decigrams of an alkali, exactly the 100 measures of the acid solution would be required to neutralise it; and if the alkali contained any impurity, such as water, carbonic acid, or a neutral salt, so much less of the acid solution would be required, and the quantity of the acid left after neutrality had been attained would indicate the percentage of impurity in the alkali. If one-half of the sample was impurities, there would be one-half of the acid left. If the sample contained only 5 per cent. of impurity, 5 measures of the acid solution would be left, and so on.

For convenience, it is usual to prepare much more of the solution than is used for one operation, the solutions thus kept in readiness being commonly called normal solutions, or volumetric solutions. 63 grams of oxalic acid, dissolved in as much water as makes up the measure to 1 litre or 1000 cubic centimetres, constitutes the volumetric solution of oxalic acid of the British Pharmacopœia.

To facilitate the making of volumetric solutions, a graduated bottle is used for dissolving the reagent, or mixing solutions which are too strong with the requisite quantity of water. For the oxalic acid solution, nothing more is necessary than to put the 63 grams of crystals into the bottle with sufficient water to dissolve them, and when solution is complete, to add water till the whole measures accurately 100 cubic centimetres or one litre.

The cut represents the usual form of graduated bottle for this purpose. You will find other volumetric utensils and

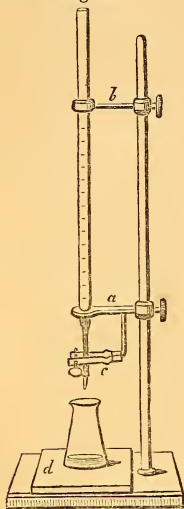
Fig. 42.



apparatus described and figured in Griffin's "Chemical Handicraft."

The next cut represents the burette and stand. The graduated tube may be compared to an elongated glass syringe with a piece of soft india-rubber tube drawn over the point, and an additional glass point attached to the lower end of the rubber tube; between these two glass points is placed a spring clip or pinchcock *c*, which prevents the flow of the liquid from the burette except when the spring is eased by the finger and thumb of the operator. The arms *a*, *b*, move upon the upright rod of the stand, to facilitate the adjustment of the burette to any convenient height, according as a beaker or evaporating dish may be found more suitable for the solution in hand; a white dish showing a change of colour most distinctly, but a glass vessel being better adapted for observing the formation of white precipitates.

Fig. 43.



The white slab *d* is convenient for testing the oxidation of iron solutions as described in the Pharmacopœia under saccharated carbonate of iron, &c., a number of little spots of red prussiate of potash solution being placed upon the slab before commencing the operation; when the bichromate solution is run from the burette into the beaker, and a drop of the iron solution taken out from time to time with the stirring rod and let fall upon one of the drops of red prussiate, the action being complete when Prussian blue is no longer produced.

The burette should in the first instance be filled above the top graduation, and the excess allowed to run out by loosing the pinchcock, as it is much easier thus to get the level of



the liquid exactly at the graduation, and what is of more importance, it also insures the whole apparatus being filled down to the delivery point, and avoids the error which would necessarily arise if this were not the case.

The following table shows at one view the volumetric solutions of the Pharmacopœia expressed in English weights and measures; but we have only to read cubic centimetres instead of grain-measures, and grams instead of grains, to convert it into the metric system.

*Volumetric Solutions.*

	1000 grain-measures contain grains.	Fraction of equivalent in grains.
Bichromate of potass. $\text{KO}, 2\text{CrO}_3 = 147.5$	14.75	$\frac{1}{10}$
Hyposulphite of soda, $\text{NaO}, \text{S}_2\text{O}_2 + 5\text{HO} = 124$	24.8	$\frac{1}{5}$
Iodine, $\text{I} = 127$	12.7	$\frac{1}{10}$
Nitrate of silver, $\text{AgO NO}_5 = 170$	17.0	$\frac{1}{10}$
Oxalic acid, $2\text{HO}, \text{C}_4\text{O}_6 + 4\text{HO} = 126$	63.0	$\frac{1}{2}$
Caustic soda, $\text{NaO} . \text{HO} . = 40$	40.0	1

In the case of caustic soda an exact equivalent is taken; but as this material is not conveniently prepared and kept in a state of sufficient purity to be weighed out in this way, it is prepared from a solution which is known to be stronger than enough; estimating its strength by volumetric examination, with the oxalic acid solution, and subsequently adding water to obtain the exact composition required. The Pharmacopœia gives a somewhat complicated calculation to show the dilution required in a given case. For illustration, 930 grain-measures are supposed to neutralise 63 grains of pure oxalic acid, and the calculation is to show how much water is to be added to 9000 measures of this strength to reduce it so that 1000 grain-measures shall be neutralised by 63 grains of oxalic



acid, the formula being  $9000 \times 1000 \div 930 = 9677$ . The 9000 measures of solution of soda being put into a graduated measure, is to be filled up with water to 9677. The simpler mode being thus—The measure of 930 grains is found to contain as much soda as ought to be in 1000 grain-measures; therefore take 930 measures, and fill up with water to 1000, and the desired result is obtained.

When we have to examine a definite chemical compound by volumetric analysis, and the reaction is of such a simple nature as the neutralising of an acid or an alkali, it is most convenient to operate upon the number of grains or decigrams which would, if the chemical under examination were pure, require 1000 grain-measures or 100 cubic centimetres of the volumetric solution to complete the reaction, because in that case the number of measures of solution left in the burette at the moment the action becomes complete, indicates the percentage of impurity the sample contains. In various cases this is the official instructions; but it is not conveniently applicable in several of the solutions, such as potass, soda, and lime waters, though it appears to me that it might have been advantageously made more general than it is. For example, in the case of vinegar, of which we are directed to use 445·4 grains, which quantity is expected to be neutralised by 402 grain-measures of volumetric solution of soda, we might equally well use 1108 grains, in which case 1000 grain-measures ought to be required, and any residue of soda solution on the burette after neutrality had been reached would indicate the excess of water in the vinegar; if 57 grain-measures were left, we would consider that the vinegar had been adulterated with water to the extent of 5·7 per cent. The object of using 445·4 grains is to enable the operator to measure it with an ordinary ounce measure, this being the weight of a fluid ounce; but pharmaceutical measures scarcely admit of the quantity being estimated with the accuracy desirable; either weighing, or the use of such measures as are made especially for volumetric purposes, should be preferred, and this would have the

advantage of facilitating the conversion of the quantity required for the operation with English weights and measures to the corresponding quantity in the metric system. If the 445·4 grains are to be weighed, the change is perfectly simple, namely, take 44·54 grams ; but if they are to be weighed, why take this odd number, including a fraction ? If we are to estimate it by measure, it would be more likely to be done accurately by taking 1087 grain-measures of vinegar to be neutralised by 1000 grain-measures of the soda solution, and then any one who might habitually use cubic centimetres would simply take 108·7 cubic centimetres to be neutralised by 100 cubic centimetres of soda solution, and the two measurements would naturally be more likely to correspond accurately with one another than would be the case if a druggist's ounce-measure were used for the vinegar and a burette for the soda solution.

In operating upon solids it is usual to weigh an equivalent in grains, or such fraction of that number of grains as corresponds with the volumetric solution, taking into account the number of equivalents that may be involved in the decomposition.

Under the tests for carbonate of potass we are directed to take 83 grains for the operation, which should require *at least* 980 grain-measures of the volumetric oxalic acid for neutralisation ; the fact being that it should require 1000 measures, but a little latitude is reasonably allowed, as the mode of its production is intended to leave 16 per cent. of water combined with it, and it readily absorbs more if not carefully guarded. The use of only 980 grain-measures of the acid solution would indicate that the salt contained 2 per cent. more water than the theoretical quantity, or an equal quantity of some other impurity.

Under the head of volumetric solution of bichromate of potash, we find four preparations of iron are to be tested with it, and the quantity directed is 20 grains of salt, requiring diverse quantities of the chromate solution ; and if the quantity actually needed is less, the deficiency does not so readily convey an idea

of the proportion deficient. The following table shows the same testing calculated for the use of such weights as should require 1000 grain-measures of the bichromate solution :—

		Grains.	Grain-measures.
Ferri arsenias	. .	117	1000
carb. sacch.	. .	60	1000
oxid. magnet.	. .	240	1000
phosphas	. .	80	1000

Used in these quantities, every ten grain-measures not required indicates one per cent. of deficiency in the substance under consideration. And if the quantities of magnetic oxide and arseniate seem inconveniently large, half the quantity might be taken; in which case, the deficiency of five grain-measures would indicate one per cent. deficiency in the proportion of proto-salt present.

## LECTURE XXVI.

### PHARMACY OF SPECIAL DRUGS.

#### CINCHONA.

BESIDES the study of the chemical and physical forces which form the base of the pharmaceutical edifice, and the processes of official and extempore pharmacy which constitute the ordinary superstructure, there is yet another addition to the building which may be considered the pinnacle of pharmaceutical science, and consists of such a general and critical view of all that we have learned as will enable us to detect the failings in any process, or in the special application of any process, and devise improvements in the same. This task is one for which the tyro in pharmacy is necessarily unqualified, and which, when undertaken even by those who have grown old in the art, forces upon them the consciousness of the vast unexplored. I would wish you to feel that in treating this part of my subject I do it purely in the spirit of a student. I speak not as one having authority, but as one who, having made some progress in his subject, is feeling his way to further advances.

You might perhaps ask why I, occupying as I do the position of a teacher, should come before you proposing to treat of problems upon which I cannot speak authoritatively; and I might reply that I do not know any lesson of greater value that I could give you than inculcating by my words, and illustrating by my example, the spirit of an investigator in the field of practical pharmacy. I propose to draw your attention to the direction and manner in which I think the pharmacy

of one or two drugs might be advantageously studied, pointing out the bearing of any recent experiments, and the direction in which further experiments seem desirable.

We may lay down as axioms that the product must have—

1st, The right kind of therapeutic or physiological action.

2d, Uniformity in degree of activity.

3d, Permanence in value.

4th, It must be obtained with as little cost of time, labour, and material as practicable in carrying out the previous requirements.

Turning our attention first to cinchona, we have to consider—What are the preparations of cinchona, official and non-official, and how far do they meet the requirements of the axioms just laid down.

In the British Pharmacopœia the official preparations from cinchona are—

Infusion, Decoction, Tincture, Fluid Extract, Quinine Sulphate.

The citrate of quinine and iron, and the aromatic iron mixture, do not come under the head of cinchona preparations for our present purpose.

The non-official preparations named by Squire are—

Compound cinchona mixture of Charing Cross Hospital, containing decoction of bark and carbonate of ammonia; and an acid cinchona mixture of St Thomas' Hospital, containing decoction of bark and sulphuric acid.

In Beasley's formulary we find—

Extract of cinchona, L., extracted with water.

Extract of cinchona, E., extracted with spirit and with water.

Extract of cinchona, dry; Paris Codex; by percolation with water and evaporation to a scaly condition.

Extract of cinchona, dry, Prussian, by macerating yellow bark in water, evaporating to a small bulk, filtering, and continuing the evaporation to dryness. This is said to contain little quinine.

Extract of cinchona. Resinous, L. 1809 and 1788, by extraction with spirit and evaporation to dryness.

Infusion of cinchona with lime water, U.S. The bark macerated in cold lime water.

Infusion of cinchona with magnesia, by boiling with magnesia, and straining.

Liquor of cinchona, Battley's preparation, very similar to the official liquid extract.

Syrup of cinchona. A variety of preparations under this head—none of any moment.

Tincture of cinchona, Huxham, only deviating from the ordinary in being prepared with brandy.

Tincture of cinchona, ammoniated, by macerating bark in aromatic spirit of ammonia.

To these we may add—

“Hospital quinine,” or “unbleached quinine,” which is simply sulphate of quinine containing colouring matter and a variable amount of other impurities.

“Quinovia,” a preparation lately introduced by Messrs Southall, containing the whole of the basic constituents of cinchona bark, minus the woody fibre.

Liq. quinae ammoniatus, in which quinine is held in solution in proof spirit with ammonia.

Syrup of dikinate of quinine, one drachm of which contains 2 gr. of dikinate, and is said to be equal to  $3\frac{3}{4}$  oz. of decoction of bark, or 96 gr. of powdered bark.

In this list sulphate of quinine stands pre-eminent. Its therapeutic action being so nearly the same as that of the entire bark, that we may say when cinchona is indicated quinine is equal to the occasion, and that it has the advantage in the second requirement, viz., that while bark is variable the quinine is constant; and this advantage is one that it holds equally when brought into comparison with the galenic preparations of the bark.

As regards permanence, I do not know that we can place it

above cinchona itself if kept dry, but we have good reason to give quinine a preference over most of the galenical preparations.

Then, as regards the question of cost,—if we take the commercial value of quinine (not sulphate) as 12s. per oz., and yellow bark, containing 2 per cent. of quinine, as 5s. per lb., we find—

$$1 \text{ lb.} = 7000 \text{ grains} \times 2 \div 100 = 140.$$

Thus, 140 grains of quinine in the form of bark costs 5s., but if bought as the pure alkaloid,—

$$437 \text{ grains} : 12\text{s.} :: 140 : 3\text{s. } 10\text{d.},$$

the same quantity only costs 3s. 10d., taking the prices at present paid by ordinary retail pharmacists. We therefore find that quinine and its sulphate fulfil also the fourth axiom I have laid down, even when brought into competition with the raw material; and, if we make a similar comparison with any of the galenical preparations, the advantage in favour of quinine is still more conspicuous.

Why then, we may ask, is not the alkaloid exclusively used? To discuss this question with advantage we should require more medical knowledge than pharmacists are expected to possess; but it does not require any great penetration to see that true progress in medical science must be promoted by the use of such remedies only as possess in a great degree the requirements of my second and third axioms, and it does not require any great experience in pharmaceutical matters to see that quinine is gradually driving other preparations of bark out of use. I do not mean to say that other cinchona alkaloids will not hold their place or even grow in importance. I have spoken of quinine as the official alkaloid, and am speaking of it in comparison with preparations the value of which depends in great measure upon the quinine they contain; but my remarks will in all probability apply with nearly equal force to pale bark and its alkaloids, or any other phase of the



question where pure alkaloids and definite salts of the same are brought into comparison with such preparations of the bark as naturally possess the variability of the raw material, and, added to that, will frequently involve a further variability, depending upon the liability of the preparation to undergo change by keeping.

The pharmacist's duty in relation to the preparations of cinchona will be mainly confined to the testing of the raw material, the examination of the efficiency of the extraction it is subjected to, and the examination of his preparations from time to time to guard against deterioration by keeping, and by this means he will probably aid in the improvement of some processes, promote the expunging of others, and in his small measure expedite the coming of that day when the application of remedies will be reduced to a scientific system.

The experiments which form the basis of my present lecture originated in the endeavour to produce a concentrated decoction of yellow bark, which, when diluted with seven times its bulk of water, would produce a better representation of the official decoction than the samples I had met with in commerce, which I had always found deficient in bitterness.

As a preliminary experiment, decoction of bark was prepared exactly according to the official formula, the degree of comminution being regulated by a sieve  $24 \times 24$  wires in the square inch, and of course contained much fine powder as well as the rough.

The decoction obtained was pale brown and turbid; the marc was boiled with a second quantity of water in the same way, and produced a decoction which was darker than the first, but less bitter, in about the proportion of one to four; a third decoction still extracted colour freely, but very little bitterness. The official decoction was reserved for a comparison with a concentrated preparation obtained as follows:—

Five oz. fine powdered cinchona was boiled with 10 oz. water (this being eight times the proportion of the British Pharma-



copcea), the whole put in a percolator; the liquor came through nearly bright and very pale, cold water was added, a little at a time, till 5 oz. had percolated; it passed through very slowly (about fl. 3j. per hour),  $4\frac{1}{2}$  fluid ounces of rectified spirit being poured in small portions at a time upon the marc; when this had all sunk in, the percolation was continued with water. The first 10 oz. of percolate ("A") contained little spirit, and was pale, with a little sediment; the second 10 oz. ("B"), which was kept separate, was brown, with a bulky light sediment, due to the resinous matter taken up by the spirit being separated again by mixture with the aqueous portion of percolate which went to make up the 10 oz. The first, "A," and two samples of concentrated decoction of cinchona from London firms of good standing, were then compared with the official decoction, one drachm of each of the concentrated preparations being mixed with 7 drachms of water. They all differed from the official decoction in appearance, being bright instead of milky, they were also all less bitter and astringent, but "A" was the best of the three.

"A" and "B" were evaporated in a water bath to 10 oz., the sediment they contained dissolved as the liquor became hot, and deposited again on the residuary liquor becoming cool. While new, one fluid drachm of this liquor with 7 drachms of water very much resemble the official decoction in colour, turbidness, and taste.

In this experiment we may note that the bark was finely powdered, boiled in water, percolated with cold water, then with spirit, and again with cold water, the percolation being so slow as to extend over nearly a fortnight; and yet, when a few ounces more water were poured upon the marc, the percolate had as much bitterness as the official decoction, showing that it was far from being exhausted. As it was not expected that the liquor would keep well without spirit (the spirit used in percolation having been driven off by the evaporation), the evaporation was continued with the addition of the few ounces of percolate subsequently obtained, the whole being reduced

to eight ounces ; as this refused to pass through filtering paper, it was set aside for a fortnight, during which time the sediment became granular, and the liquor began to turn mouldy. It now filtered without difficulty. The sediment in the filter was in great measure dissolved by passing  $2\frac{1}{2}$  oz. of rectified spirit through it ; when the whole had passed through, the filtrate contained 25 per cent. of spirit, and was clear and brown, and did not become milky on dilution with water, but was found to deposit a brown sediment on keeping ; after the lapse of several months, it still continued to give a good representation of the official product, excepting that it was bright if decanted, and if shaken gave a brown liquor, not having the milky appearance of a fresh decoction.

I have described this experiment somewhat in detail, as it was not very long or complicated, and led to one conclusion which you may regard as having some practical interest ; but it still left untouched the points which have a deeper importance than simply providing a trade formula.

The questions next to be considered were the proportion of active matter left in the marc, and the practicability of extracting them in such a manner as to make them available for use as decoction or infusion of bark. The experiments evolved from this consideration were much too lengthy to be quoted in detail, but we may find advantage in running over the results, and taking a little closer note of one or two of the more important.

Ten oz. of powdered bark packed moist in a percolator, and water percolated to 80 oz., the last portion of the percolate was very pale and had *little bitterness* ; the contents of the percolator, without being disturbed, were then supplied with 1 oz. diluted sulphuric acid and 3 oz. water mixed. When this had sunk in, percolation with water continued. As soon as the percolate began to be acid, it was again *strongly bitter* ; as soon as this percolate amounted to 80 oz. the last portions were found to have only the small degree of bitterness which corresponded to the last portion of the first 80 oz., the totals of second percolate being more bitter than the total of the

first, showing that the acid much increases the power of water to extract the quinine from the bark. The marc still remaining in the percolator was again treated with acid and water, and again as soon as the percolate became *slightly acid*, it was also *strongly bitter*. This third percolate was continued up to 140 oz., which on evaporation was found to contain less bitterness as a total than either the first or second.

A fourth percolate was obtained in a like manner and with similar results, except that the bitterness was evidently diminished. This percolate amounted to 90 oz., and on being evaporated was found to have less than half the bitterness contained in the first 80 oz. percolated.

We have noticed above, that when the bark had almost ceased to impart bitterness to water, that acid extracted bitterness freely; that the bitterness disappeared as the percolate ceased to be acid, and reappeared as it became acid again from a fresh supply; and we have now to add to these observations that 19 pints of water and acid together, slowly percolated through 10 oz. of finely powdered bark, failed to effect complete exhaustion.

This does not encourage us to expect that we could obtain a satisfactory exhaustion of the bark by the official process for liquid extract of cinchona, the Pharmacopœia instructions being to percolate 16 oz. of roughly powdered bark with 12 pints, or a sufficiency of water.

I have not had experience in the making of the official liquid extract, but from the examination of a reliable sample, it appears to me that about the same degree of exhaustion had been obtained as I obtained by percolating 80 oz. of cold water through 10 oz. of fine powdered cinchona. My finished product of 20 oz. from 10 of bark (without acid) was  $\frac{1}{3}$ th the strength of the British Pharmacopœia liquid extract, where 4 fluid oz. are obtained from 16 oz. of the powder, showing that if the samples of bark were equally good, the extraction by the British Pharmacopœia process had only gone as far as mine had gone before I used acid.

Another point ascertained was, that these percolates containing sulphuric acid could be rendered neutral without loss of bitterness by adding an excess of chalk, and afterwards filtering to separate the sulphate of lime and undecomposed chalk.

Again, we must note that 10 oz. of powdered bark should by calculation yield 1 pint of concentrated decoction for diluting 3j. to 3vij., but that the product obtained was nearly  $3\frac{1}{2}$  times that quantity of liquor of the required degree of bitterness.

In two other experiments the powdered bark was rubbed with sulphuric acid and water, and allowed to macerate before the percolation was commenced. In the first case the liquor was only added in sufficient quantity to reduce the 10 oz. of bark to a moist powder, and left thus eight days, when it began to turn mouldy; percolation was then commenced with water only, and followed up with water and acid. 8 or 10 pints failed to exhaust the bark. The yield equalled 3 lb. of concentrated decoction.

In the second case the bark was mixed with 28 oz. of weak acid, and allowed to macerate five months, during which time three separate films of mould had formed on the surface of the liquor, and then sunk down. It was then submitted to percolation, the sourness of the percolate rapidly decreased, but its bitterness was not notably diminished when 6 pints had percolated. This being evaporated and neutralised, equalled 1 pint of concentrated decoction. The second 6 pints of percolate, evidently not so bitter, were unfortunately lost, but not before it was noticed that the last portion of it was but slightly bitter. The third percolate was commenced with acid, which was allowed to remain in contact with the bark for two days before more water was added to drive it through; when it came through it was darker, and only slightly more bitter than the neutral portion which preceded it. From this we note that the long maceration with acid had brought the alkaloids into a condition in which they could be entirely, or almost entirely, washed out with water, but had at the same time

diminished the quantity of bitter obtainable, the loss probably arising from decomposition which had taken place in consequence of the moulding, or perhaps it would be more prudent to say in consequence of the long keeping, for the moulding may be a coincidence, and not a cause.

In comparison with these, two similar experiments were tried with acetic acid instead of sulphuric.

In the first of the two the acetic acid and water was used to moisten the powder, and no mouldiness appeared in 24 days; but I must also add that this long contact of the acid had not brought the alkaloids into a condition to be satisfactorily washed out with water, for the bitterness of the percolate, when greatly diminished, was renewed on percolating acidulated water; and this occurred with a *third* portion of acetic acid. When the exhaustion was thus approximately complete, the yield of bitter principles was estimated as equal to 2 lbs. of concentrated decoction; against 3 lbs. obtained by the use of sulphuric acid.

In the second acetic acid experiment the quantity of liquor being 25 oz. to 10 oz. of bark, and the maceration six months, no mouldiness occurred. This being transferred to a percolator, and when drained water poured upon it, the percolation was very slow, and the percolate was much less bitter by the time 3 pints had passed through. This, on evaporation to 1 pint, had scarcely the bitterness of the standard concentrated decoction of cinchona. The marc, when percolated with acid, did not again yield a *strongly* bitter solution, and the liquor became mouldy during the percolation, apparently losing part of its bitterness at the same time, the proportion of acetic acid present being too small to act as an antiseptic. The total yield of bitter principles is less in this than in any former case. Apparently the long maceration, even when not accompanied by moulding, results in a loss of alkaloids.

The next experiment was designed to ascertain if the bark could be satisfactorily exhausted, by treating it first with spirit, which it was supposed would extract the resinous matter, and

thus remove the one impediment to the dissolving of the alkaloids. 1 lb. of finely powdered bark was treated as for the British Pharmacopœia tincture, the percolation was very slow, occupying about a week, and the exhaustion might be supposed to be more thorough than is usually effected in the making of the official tincture. The marc, after pressing, retained about 12 oz. of spirit. It was rubbed down with 8 oz. of water, placed in a percolator, and more water added as the dropping ceased till 40 oz. were obtained, which were still bitter and tasted of spirit; the taste of spirit disappeared when about 50 oz. had percolated; the bitterness of the last portion was about equal to a solution containing  $\frac{1}{20}$  grain of sulphate of quinine in 1 oz. of water;\* the bitterness of the whole 50 oz. when mixed was about equal to a solution containing  $\frac{1}{6}$  gr. sulphate of quinine in 1 oz. of water.

The percolation being continued with sulphuric acid and water, again became strongly bitter, about 35 oz. percolated, the average bitterness of which was about equal to a solution containing  $\frac{3}{8}$  gr. of sulphate of quinine in an ounce of water. We have here to note *that the successive action of spirit and water does not readily exhaust the bark*. As the percolate was beginning to turn mouldy, the percolation was continued with a mixture of proof spirit 9 oz., diluted sulphuric acid 1 oz.; when this had sunk into the marc, water was again poured on till 80 oz. of percolate had been obtained, the bitterness of which equalled a solution of  $\frac{3}{8}$  gr. of sulphate of quinine in 1 oz. of water. We must now add to the above note, *that extraction of the bark with spirit first does not leave it in a condition easily exhausted by percolation of either water or water and sulphuric acid*.

Percolation now became more rapid, and was continued with acid and water till a further percolate of 110 oz. had been

\* I do not regard these comparisons as giving any accurate idea of the quantity of quinine contained in the percolate, but I give them, as the only ready mode of conveying an idea of the comparative degrees of bitterness observed in the various portions of the percolate.



obtained, having a bitterness equal to  $\frac{1}{8}$  gr. sulphate of quinine to an oz.

The last 10 ounces of the above was so nearly free from bitterness, that the bark was considered practically exhausted ; this was confirmed by allowing the acidulated water which it retained by capillary attraction to remain there for a fortnight, and then displacing it with more water, when it passed through slightly acid, but almost free from bitterness.

The time and solvents used in exhausting this pound of finely-powdered cinchona were as follows :—

	Days.	Pints.
Rectified spirit, . . .	...	4
Macerated and percolated, .	10	...
Water, . . .	...	$2\frac{1}{2}$
Percolated, . . .	14	...
Water with acid, . . .	...	$1\frac{3}{4}$
Percolated, . . .	10	...
Water with acid and spirit, . .	...	4
Percolated, . . .	28	...
Water with acid, . . .	...	5
Percolated, . . .	15	...
	<hr/> 77	<hr/> $17\frac{1}{4}$

It has long been known that the ordinary infusion and decoction of bark failed to extract anything like the whole of the alkaloid; indeed, Whittstein, in his article on the Manufacture of Sulphate of Quinine, says,—“The residue from aqueous infusions contain most of the quinine, and may be advantageously employed by pharmacutists.”\*

Even the process by which we are directed to estimate the quinine in finely-powdered bark can only be considered approximate; for example, I may quote the examination of the same sample of powdered bark which supplied the material of most of the experiments already quoted :—100 grains in fine powder were boiled in 1 ounce of water

\* Whittstein's “Practical Pharmaceutical Chemistry,” translated by Stephen Darby.—A little book of great value to the student who wishes to understand the chemistry of pharmacy; though it has come of age, and is of foreign extraction, it ought to be the apprentices' companion.

acidulated with hydrochloric acid, decoction and marc poured into a small percolator, and when drained percolated with, first,  $1\frac{1}{2}$  oz. of acidulated water, and then with another ounce, the percolation extending over three days; it was then considered approximately exhausted, and the analysis completed on that supposition; but for experiment's sake the percolation of acidulated water was continued through the marc, and still extracted a little colour and bitterness, even after the lapse of a week or more. The quinine was not estimated in the second portion of percolate. That in the first weighed 2·8 grains, and was not quite free from colour.

No one could regard his analysis as quite satisfactory even for pharmaceutical purposes; if the marc was known to contain a portion, and though we may say a small portion, we must also admit that it is an uncertain proportion of the alkaloids; and as even this imperfect extraction was affected by the expenditure of four days—one for maceration after boiling, and three for percolation—no one is likely to undertake the task as a check upon the quality of the few pounds of bark, which many retail pharmacists will buy at once; and unfortunately, the after part of the analytical process is not so simple in practice as to tempt us to use it as a method of examining the quinine value of our pharmaceutical preparations of cinchona.

This concludes the experiments on extraction as far as I have carried them. I was desirous of finding a suitable mode of extracting the whole of the active matters without the necessity of using both acid and heat (as practised in the quinine manufacture), and so far I have failed, but not without some instructive results.

Prior to the commencement of my experiments, I felt disposed to recommend the discontinuance of either the decoction or infusion of cinchona, on the ground that it is unnecessary to retain two preparations which are identical in all respects but the method of their production; I now feel more disposed to advise that both be rejected, and that, if it be thought



desirable from a study of the action of cinchona and its alkaloids, to continue the use of any preparation containing the mixed constituents of the bark, that the tincture might be retained, and either the liquid extract or a similar preparation obtained by some improved method not yet elaborated.

The loss of bitterness resulting from evaporation is considerable, and the fact has an important bearing upon the value of the extracts of cinchona; I must quote in detail one experiment upon this point.

Six ounces of British Pharmacopœia decoction, evaporated in water bath to dryness, left a brown residue, which only partially dissolved in water; diluted to 6 oz. with distilled water and strained through tow, gave a liquor rather browner than the fresh decoction, and having about two-thirds the bitterness and astringency.\*

The brown matter not dissolved by water was almost entirely dissolved by rectified spirit, forming a brown solution which became turbid on a large addition of water, but did not represent all the bitterness and astringency which the watery liquor had lost.

The watery liquor, being filtered through paper, was bright and almost colourless; it then measured  $5\frac{1}{2}$  ounces (the loss being the portion consumed in tasting and that absorbed by the filter); it was then evaporated down again to 3 fluid drachms, and filtered to separate the brown colouring matter which had again deposited; when diluted with the washings of the filter to  $5\frac{1}{2}$  ounces, it possessed about one-half the original bitterness; a third evaporation still caused the separation of colouring matter (probably the *production* of colouring matter by the combination or decomposition of bitter and astringent principles); the evaporation having been carried to dryness, the residue treated with water and filtered, yielded 5 ounces of clear pale liquor, having only one-third the original bitterness.

\* The strength in these experiments was estimated by ascertaining how much water was required to be added to a portion of the original decoction to reduce it to the same taste as the liquor under examination.

This leaves little doubt that repeated evaporations would ultimately reduce the whole of the extract to the insoluble condition, and probably destroy its medicinal value; and it must be noted, that the evaporation which was not carried to dryness was not free from this injurious action. I have noticed the same loss of bitterness in other cases where neutral cinchona liquors were evaporated to small bulk, but in several cases, where the liquors contained free sulphuric or acetic acid, this loss did not take place. We may in this connection again note, that the dry extract of cinchona of Prussian pharmacy "is said to contain little quinine," and it would be interesting to have observations by other pharmacists relative to the comparative bitterness of the liquor for liquid extract of cinchona before evaporation, and a portion of the finished extract diluted with water to an equal bulk.

It seems probable that evaporation to so great an extent may cause a considerable loss of alkaloids, and it becomes a problem whether the preparation could be improved by acidulating the liquor previous to evaporation, say with sulphuric acid, and subsequently removing this by neutralising with chalk.

I have also noticed in various experiments that the absence of colour, or the approximate absence of colour, to speak more critically, did not indicate exhaustion of the alkaloids, nor did the more free extraction of colour indicate that bitter principles were being dissolved. Note the first experiment quoted.

We may consider that all the galenical preparations of bark are faulty as regards extraction, and our next question is, have any of them special advantages to counterbalance this fault?

We hear of medical men preferring decoction of bark and carbonate of ammonia as a tonic to fever patients; but the solubility of quinine in the presence of ammonia, though not great, is sufficient to enable as large a quantity to be given in solution in water with ammonia as is contained in the decoction of bark, and its solubility in ammoniacal spirit is very considerable; the ammoniated solution of quinine, quoted in

my list of non-official preparations, and the ammoniated tincture of cinchona, P. L. 1824, would probably supply all that need be provided in this direction.

These preparations suggest the inquiry—Does ammoniacal spirit dissolve the quinine so freely as to be a better solvent for extracting the bark than any of those at present in use? Lime-water also dissolves quinine more freely than pure water, and it was no doubt on this account that the infusion in lime-water was introduced into American pharmacy, and probably the absence of advantage led to its subsequent rejection. Lime-water might dissolve quinine more freely than pure water, and yet might not dissolve quinine more readily than water dissolves kinate of quinine.

In Storer's "Dictionary of Solubilities," under the head of quinine, we find its solubility given as 1 part in 350 of water, but that its solubility is much greater in various other solvents; for example, 1 part in 2 of alcohol, in 60 of ether, 2 of chloroform, 24 of olive oil, &c. It is also stated that kinate of quinine is readily soluble in water, and less so in spirit. We also are well aware of the free solubility of quinine and most of its salts in many acid liquors, and yet we have seen the exceeding difficulty in getting it thoroughly extracted from the bark, and our observations have not led to any explanation of the cause of this difficulty nor the way in which it could be satisfactorily overcome. So long as this is the case, it seems too much to expect the small pharmacist to submit his cinchona to the tedious process of analysis, which, after all, will only give him an approximate indication of the quantity of quinine his bark contains, and no indication of how much it is likely to yield when submitted to any of the official processes, excepting the one he cannot perform for himself, viz., the manufacture of sulphate of quinine.

The exceeding solubility of quinine in alcohol, and the comparatively small solubility of kinate of quinine in the same solvent, taken together with the powerful action which this solvent has upon resinous, astringent, and colouring matters of

the bark, suggests that extraction *might* probably be effected more readily by the joint action of spirit and caustic soda, potass, or ammonia, these reagents adding to the power of the spirit to dissolve all the matters which may be supposed to impede the solution of the alkaloid. If thorough exhaustion were thus readily effected, it would still leave the problem of reducing the liquor into a condition suitable for administration or for yielding the alkaloid in a pure form.

It also appears not unlikely that extraction might be more readily and thoroughly effected than by any of the present official methods, by the joint action of spirit and acid, say a long maceration of fine powdered bark in spirit and sulphuric acid, followed by percolation of water, evaporation to any required bulk, neutralisation with chalk, and filtration to yield liquor of cinchona less wasteful than the present decoction.

It is worthy of an experiment to ascertain if maceration of the bark in acetic acid and spirit, followed by percolation with water, and evaporation to so small a bulk as to remove the bulk of the acetic acid, would yield an acetic extract, either fluid or semi-solid, which would give a fair representation of the active matters of cinchona.

The loss of bitterness observed in several of the preparations by keeping, and the loss observed during evaporation of the decoction, coincident with the appearance and precipitation of colouring matter, suggest the need of experiments upon the stability of official cinchona liquors, there being grounds for a reasonable fear that the brown deposit which takes place in the liquid extract of cinchona may indicate a loss of alkaloids, even though the deposit may not be found on analysis to contain any.

For the pharmacist to exercise a constant supervision over his preparations, it is desirable to have some means of comparison less tedious than that now adopted, of separating the quinine. I have made examinations of this kind by taste, which I think is capable of affording comparisons close enough for pharmaceutical purposes when the alkaloid in the standard

of comparison is accompanied with the same principles,—acid, astringent, or otherwise, which are present in the sample under examination, and the precaution is taken of having the solutions considerably diluted, and making the estimate by ascertaining how much the stronger of the two requires dilution before they taste equal. It is not unlikely that further experiment may indicate a convenient standard, and the means of readily bringing the samples for comparison into a similar condition.

I have also observed the degree of fluorescence in several cases, but not with sufficient care to warrant a prediction of any results from this mode of comparison; it is, however, an interesting subject to pursue.

The subject has grown much longer under treatment than I at first anticipated. My cinchona tree has produced many branches and but few fruits. I present it to you now, not as perfected, but as growing, and if the requisite leisure be at my disposal to attend to it, the branches which are now blossoming may be bearing fruit at another season.

## LECTURE XXVII.

### PHARMACY OF SPECIAL DRUGS.

#### OPIMUM.

THE principal points which claim our attention in connection with the pharmacy of opium are somewhat different from those which were considered most important in the treatment of cinchona. Though the complete exhaustion of opium is not very readily effected, the official processes all afford a fair approximation to this result. In making a preparation of cinchona, the principal object was to separate the bulk of inert matter; in making an extract of opium, using the word extract in its wider sense, our aim is to separate matters which are considered to have an objectionable action, and to retain one or more principles which possess beneficial properties. In both cases an important object is to insure uniformity of strength.

There are many of the official preparations of this drug which it is unnecessary for me to comment upon; the remarks I may have to make upon the raw material will have a bearing upon the different preparations containing the crude drug or its powder, and anything affecting the quality of the tincture or extract will in like manner influence the secondary products, in the production of which these primaries are used.

Two pounds of opium in its crude state yield by the official process about 1 pound of extract of a pilular consistence, the yield depending upon the dryness and amount of soluble matter which the sample contains; in one instance in which I obtained this yield, I also noted that the residue, I must not

say insoluble residue, but that which was not dissolved by this treatment, yielded on analysis about 10 gr. of morphia to each pound of the original opium, which had been weighed in its moist condition, and in that state contained about 7 per cent. of morphia. This indicates a good practical exhaustion. The pound of opium would contain 490 grains of morphia, of which 480 would remain in the half pound of extract yielded by the process.

A similar experiment upon the marc of tincture of opium indicated about 9·3 grains of morphia remaining in the marc for each pound of opium originally used. This would imply that spirit produced a more thorough exhaustion of the opium than water, for though the quantity of spirit used to extract 1 pound of opium in making tincture, is  $10\frac{1}{2}$  pints against 6 pints of water used for the same quantity of opium in making extract, the mode of operating in the latter case might be supposed to do more than compensate for the smaller quantity of solvent used. The action of a solvent in successive portions is usually much more effective than it is when the same quantity is used all at once, and the three applications of pressure alternated with the rubbing into a pulp, would, in theory at least, imply a very thorough extraction of all that the water is capable of taking up. This difference appears the more striking when we note that the marc of the tincture as it came from the press retained  $\frac{1}{10}$  of the spirit used, and that portion was necessarily as strong a solution of morphia as the finished tincture, and if deducted would reduce the proportion of morphia not dissolved in the process to about one-half of that contained in the entire moist marc; while the marc from the extract, having been treated by three successive portions of solvent, would at last contain only a liquor of a considerably diluted character.

The morphia is said to be contained in the opium as a meconate, a salt which is freely soluble both in spirit and in water, but the narcotine in the absence of strong acids is but sparingly soluble in water, though comparatively freely soluble



in spirit. We may, therefore, suppose that the tincture is richer in narcotine than the liquid extract. The tincture also contains resinous, fatty, colouring, and odorous matters which are present to a much smaller extent in the liquid extract, the three former because they are less extracted by the water, and because the portion of them which is taken up by the watery liquor is again partially precipitated during its evaporation, and at the same time almost the whole of the latter, the odorous matter, is driven off. It is generally supposed that the morphia is not diminished by the process the extract goes through, though I am not aware that conclusive experiments have been tried upon this point. And if we admit that the resin and narcotine are objectionable constituents, and codeia, and other principles of a beneficial character, are present only in small quantities in the crude drug, and probably to an equal extent in the extract, there appears a remarkable discrepancy between the theoretical activity of the extract and the dose officially indicated.

Turning to the Pharmacopœia, we find the dose of opium is from  $\frac{1}{2}$  grain to 2 grains, and the dose of the extract is the same. Under the head of opium there is a table of its preparations, in which it is stated that one part of extract is obtained from two of opium, which agrees exactly with my own observations; but this being so, what has become of the activity? We should expect the extract to be double the strength, and we are precluded from supposing that the dose has been indicated the same as opium by an oversight or misprint, because the doses of the liquid extract and wine of opium are in strict accordance with those quoted for the solid extract; nor can we conclude that the variable strength of the crude drug necessitates its dose being stated as smaller in proportion than that of the extract, because, whatever variability the opium possesses, is likely to belong in equal degree to the extract. And the latitude allowed by the Pharmacopœia in the morphia value of opium, does not prevent the use of samples which vary between 6 and 10 per cent. or even more.



But we are not instructed how much morphia the extract should contain, we have no security that its variability is any less, and we have no alternative but to suppose that half the activity is lost, or that the doses are so far from being fairly and accurately compared, as to shake our confidence in the statements which are made relative to the merits of the various preparations of opium.

This opens out the first problem in the study of the pharmacy of opium; that is, to ascertain the activity of its various preparations as determined by physiological action, as compared with their theoretical value as indicated by morphimetry.

If you compare the doses of morphia with those of opium and the extract, we find that the dose of morphia corresponds more nearly with that of the extract. Morphia is about  $\frac{1}{3}$  stronger than the hydrochlorate, the dose of which is given as  $\frac{1}{8}$  to  $\frac{1}{2}$  gr. Two grains of the extract of opium may contain  $\frac{2}{3}$  of a grain of morphia, equal to little more than  $\frac{1}{2}$  grain of hydrochlorate; that is, on the supposition that the original opium contained 10 per cent. of morphia, and that as a consequence the extract contained 20 per cent.

But this involves the supposition not yet established, that the extract is prepared without the loss of any morphia beyond the insignificant portion failed to be extracted from the marc, and it is also based upon the supposition that the original opium contained 10 per cent. of morphia, and not 6 per cent., which would have been legitimately within the official standard.

From this comparison, it would appear that more morphia is given for a dose, in the form of hydrochlorate, than is given for a dose in the form of extract of opium (of average quality), and more is given for a dose in administering extract of opium than when the crude drug is employed.

All this appears exceedingly unsatisfactory. We either spend our pains in preparing articles which are wasteful of the legitimate effects of the drug, or the appreciation of the effects

of the drug and its preparation is so imperfect as to destroy all our confidence in the testimony regarding the quality or advantages of the preparations we make.

The medical side of this question is so beset with difficulties that it appears hopeless to wait for improvement till definite replies can be had to the inquiry regarding the therapeutic value of the different constituents of opium, or of the entire drug compared with morphia given in the form of any of its salts. We can scarcely get agreement or a reasonable ground for difference of opinion regarding the comparative value of these definite compounds themselves. Fortunately for us, as pharmacists, this difficult question does not belong to us. We are impelled, not only as a matter of policy, but as a matter of necessity, to trust to morphimetry indicating the true relative value of opium and its preparations, and if by chance we are mistaken in this supposition, our error can only be proved when our products have a well-defined morphimetric value, as a basis upon which the therapeutic experiments can be elaborated, and this detection of our error, or confirmation of our theory, would be the most important step in the progress of the pharmacy of opium, or the therapeutics of its proximate constituents.

Assuming, then, that the value of opium is proportionate to the morphia it contains, we may at once say that the British Pharmacopœia has made a great advance upon all its predecessors in relation to this subject, inasmuch as it has defined that opium is to be analysed before being accepted as fit for pharmaceutical use; but it has stopped short of the point at which the use of an analysis would have placed the subject in a tolerably satisfactory position, namely, the giving to opium and its preparations a fixed morphimetric value. We are told that 100 grains of opium, by the process of assay directed to be used, should yield "at least six to eight grains" of morphia. That is, it should yield at least six grains, and it is implied that seven or eight grains would be quite appropriate; but it is neither stated nor implied that ten or twelve grains

would be objectionable. But in a drug like opium it is a matter of no less importance to guard against an overdose than it is to ensure that sufficient is given. As far as the pharmacy of the question is concerned, both these points would be attained by defining that powdered opium should contain, say seven per cent. of morphia; samples above or below this percentage being brought to the standard strength by mixture with one another, and requiring that this standardised opium should be used for making all preparations except the alkaloid and its salts. Or it might be allowed, as an alternative in making the tincture, that so much opium should be used as would contain the required quantity of morphia. It would be quite satisfactory to use 7 parts of opium containing 10 per cent. of morphia in the place of 10 parts of opium containing 7 per cent. of morphia; but it is not equally certain that the use of 20 parts of opium, containing  $3\frac{1}{2}$  per cent. of morphia, should be allowed, as there might thus be introduced a much larger proportion of narcotine, which is usually regarded as an objectionable constituent. It might be a prudent addition to such regulations to exclude opium containing less than 5 per cent. of morphia except as a source of the alkaloid.

You will observe how much importance I give to a knowledge of the quantity of active principles we are dealing with. Scarcely any branch of science can be said to have taken the first step towards satisfactory progress until we are able to give numerical expression to the agencies employed, and in some measure also to the results obtained. Pharmacy and therapeutics are not exempt from this rule; I must, therefore, direct your attention next to the process for estimating morphia in opium.

Morphimetry has hitherto been a tedious operation, and I do not at present see any prospect of its ever being otherwise. The attention of many able chemists has been given to it, and I believe no process which has been published can claim any great advantage over that of the Pharmacopœia; yet I am far from willing to admit that it is incapable of improvement.

The requirements of a good process are, that it should effect a thorough exhaustion of the opium in a short time, whether the sample be soft or in powder; that it should not risk the loss of morphia after extraction; and that it should yield the morphia almost free from impurities.

The official instructions direct us to take 100 grains of opium, break it down in one ounce of water, macerate for twenty-four hours, and percolate other three ounces of water. This involves at the outset twenty-four hours, and then the time required for percolation, which varies according to the sample of opium from about twenty-four hours more to two or three days; and as the percolate continues to be strongly bitter up to the end, there is not the satisfaction of knowing that all the morphia has been extracted. The bitterness still continuing may result from narcotine, &c.; but it is more satisfactory to have further assurance that there is little or no morphia remaining in the marc.

It has been recommended to exhaust the opium first with benzine, to remove narcotine, resin, fat, and caoutchouc, the morphia being left, and subsequently more readily dissolved by water; but as this involves the drying and powdering of the opium, always a tedious operation, it is not available for the use of any one wishing to buy opium from analysis of a sample. The operation must be speedily performed, and that precludes any process requiring drying and powdering.

It has been suggested to treat the opium with lime in the first place, percolating water through the mixture, but I have found no advantage from so doing.

Nor does the use of acid appear to facilitate the solution of the morphia; it is already in a state of combination sufficiently soluble, its extraction being impeded by some other cause; it is probably mechanically protected by the fatty matters, &c., which are proposed to be removed by the benzine in the process named above. I have tried the combined action of water and ether, but there are some difficulties in operating satisfactorily with the two solvents at once, and I finally settled

upon spirit as the best solvent, the operation being performed as follows:—

Take of opium 100 grains, reduce it to a soft pulp by working it in a warm mortar with about twice its weight of water, adding more if the loss by evaporation makes the pulp stiff; when soft and free from lumps, add 2 oz. methyated spirit, and put it in a convenient vessel for subsidence and decantation; stir well, and let it subside for a few minutes; decant the clear portion, filter it, and set it to evaporate. Pour upon the marc 2 oz. more spirit, and stir several times; then let it subside, decant, filter, and add the filtrate to the contents of the evaporating dish. Treat the marc with a third portion of spirit; after this is decanted put the marc into a percolator tube and pass spirit through it, which can now be done without difficulty; continue the percolation till the percolate has but little bitterness, adding all the liquors thus obtained to the contents of the evaporating dish. This looks upon paper a more complicated process than that of the Pharmacopœia, but it may be completed in six or eight hours, including the evaporation; and the extraction is quite satisfactory, whereas the official process effects a less complete extraction by an operation which I have never been able to effect in less than two or three days.

So much for the first step in the process; the next is to separate, from the liquor obtained by either method, the morphia in a pure state. The alcoholic liquors obtained as above, when evaporated, deposit much resinous, fatty, and colouring matters; this is promoted by adding a little water towards the end of the evaporation, so that when a syrupy consistence is attained, the spirit has been entirely driven off. A further addition of water being then made, so that the whole shall measure about 1 oz., causes a further precipitation, probably consisting principally of brown resinous matter and narcotine; the liquor may be decanted from this after standing ten minutes; the precipitate, which forms a soft sticky mass, adheres to the dish, and may be washed with a small bulk of

water. The watery liquors thus obtained may be finished by the process indicated in the Pharmacopœia, unless a better can be adopted; we will now consider the next step in the official process.

The aqueous solution of opium is placed in a flask with 100 grains of slaked lime and boiled for ten minutes, the whole is then thrown upon a filter and washed with one ounce of boiling water.

The object of this proceeding is to separate, in an insoluble form, the acids of the opium, together with colouring matter and the bulk of the alkaloids except morphia; the latter being pretty freely soluble in lime-water as well as other alkaline liquors, while narcotine requires acids for its solution. But though narcotine in its pure state does not dissolve in lime-water or alkaline liquors, it is far from being satisfactorily separated in this way. The presence of organic matters frequently has a considerable influence upon the solubility of substances which would otherwise be precipitated, and the constituents of a vegetable frequently very materially aid the solution of one another; a fact made use of by Battley, who promotes the solution of the resinous principles and others not freely soluble in pure water, by taking care that the water is used in small proportion, so that its solvent power is increased by the presence of a considerable proportion of gum, acids, alkaloids, or other constituents which are normally soluble in water; and a fact which is frequently illustrated by the precipitation which takes place when a strong aqueous solution—of opium for example—is diluted with water; and so we find the narcotine which, on theory, we might expect to be excluded from the decoction with lime, is still there in considerable quantity, and is liable to be held in solution by solvents which retain the morphia and colouring matter, and precipitated by precipitants which throw them down. This circumstance very much increases the difficulty of precipitating the morphia in a pure condition.

The quantity of lime used seems larger than necessary; and



makes a great bulk of soluble matter to be washed with so small a quantity as one ounce of water. I have tried one or two experiments upon the use of smaller quantities of lime, but am afraid to give a decided opinion in favour of a reduction of the quantity.

The lime liquor is acidulated with hydrochloric acid; the effect of the first addition of acid is to neutralise the lime and cause a slight precipitate, probably of morphia and colouring matter, but when the liquor becomes distinctly acid it again becomes clear, and paler than before, the lime and morphia both being in the state of hydrochlorates; when this is the case, the liquor is evaporated to the bulk of half an ounce and cooled. It is then to be neutralised by the cautious addition of ammonia, by which colouring matter and narcotine are thrown down. This is the most critical step in the whole process, and one which stands much in need of improvement. You will readily perceive that a little too much ammonia will precipitate the morphia also, and a little deficiency leaves some of the colour and narcotine in solution, which will be thrown down with the morphia in the subsequent precipitation. The liquor being filtered from this dark precipitate, the precipitate washed with one ounce of hot water, the filtrate and washings evaporated to the bulk of half an ounce, are then to be rendered alkaline by a slight excess of ammonia, by which the morphia is thrown down, and after being allowed twenty-four hours to deposit, is to be washed, dried, and weighed. "It ought to weigh at least six to eight grains." I have never succeeded in getting morphia precipitated in this way satisfactorily free from colour and narcotine, even when the ammonia was added in such quantity as to cause the precipitation of a small quantity of the morphia along with the colour and narcotine first thrown down, and I have sought by many expedients to effect this separation of the two precipitates more satisfactorily.

The general statement, that morphia remains in solution when the liquor is quite free from acid reaction, and that

narcotine, and the brown matter accompanying it, were precipitated when the acid was neutralised, led me to hope that this might be accomplished by the addition of acetate of ammonia and subsequent evaporation to dryness. Any free hydrochloric acid in the liquor would decompose a portion of the acetate of ammonia, leaving acetic acid free, which would be got rid of by the evaporation to dryness; but the residue, when treated with water, though neutral to test paper, still contained much of the colour, a small portion only having remained insoluble. The dark brown liquor was then treated with acetate of lead without any precipitation taking place; and when again evaporated to dryness, a second small portion of colour was separated, the acetates of lead and morphia being dissolved in water; the former base was separated by the addition of sulphuric acid and filtration, the latter being precipitated by the addition of ammonia as usual, was accompanied with a large quantity of colouring matter.

In another experiment chalk was added with the view of insuring neutrality; and the liquor evaporated to dryness, failed to get rid of the colour.

The colour and narcotine, however, were found to be more satisfactorily precipitated by the addition of bicarbonate of potass to the acid solution, allowing half an hour for their deposition, filtering, washing a little water through the filter, adding hydrochloric acid to neutralise the excess of bicarbonate; the morphia is not thrown down by this treatment, and is afterwards precipitated nearly white by ammonia.

The use of spirit to retain the colour and narcotine in solution, while the morphia is precipitated by ammonia, has been advocated by Prof. Procter, and I have found it yield a brightly crystalline morphia with but little colour. It is liable, however, to be contaminated with narcotine, unless the latter body be present in only very small proportion in the original liquor.

Besides these, I have tried many other experiments upon the point which it would be tedious to describe, and what I



have done is insignificant compared to the investigations of others, the general testimony being that there is considerable difficulty and uncertainty in the operation. Bearing all this in view, I have to advocate the addition of one point to the official instructions for analysis: it is this, seeing that the precipitate will contain *some* colour, and may contain narcotine, I would say it should weigh at least seven grains, and should dissolve entirely in one drachm of water and one drachm of solution of potash, the solution being of a pale brown colour. If any portion of the precipitate does not dissolve in the alkaline solution, the weight of the insoluble portion subtracted from the whole still leaves seven grains as the weight of the morphia soluble in the solution of potash.

Messrs Dean and Brady, working together in a microscopical research into the nature and merits of the liquid preparations of opium, have shown that the microscope can give important aid in determining the comparative value of different preparations, and considerable insight into their crystallisable constituents, besides their morphia value; but their work, which is admirable for the object they had in view, cannot readily be made to serve the purpose which I am now advocating; we could scarcely define the microscopical characters or the mode of preparing the specimens for examination, with such precision as to allow of their official use to determine which samples of opium should be considered fit for pharmaceutical use, or which pharmaceutical preparations had the required strength. Even if the required degree of precision were attainable in description, the time involved, *i.e.*, several days, would place us in the same position as regards one of the difficulties of applying the present mode of analysis to commercial purposes, where the sample must be assayed within a very short time if a purchase is to be decided upon. The very beautiful plates which accompany Messrs Dean and Brady's paper in the "Transactions of the Pharmaceutical Conference" for 1864,\* are highly instructive, but still there are many ques-

\* Also in the *Pharmaceutical Journal* of the same date.

tions which we would like to put to them, and to which we can as yet obtain but an echo for a reply. We note the very great difference between the residue of tincture of opium and that of an aqueous liquor, and we are not surprised that it should be so when we remember that the tincture contains all the crystallisable constituents of the drug, while the aqueous liquors reject some of them ; but if we compare the residue of the sedative liquor, or liquid extract, with the crystallisations of meconate and sulphate of morphia, we should be disposed to doubt the presence of either of these two salts, and we ask, In what state of combination does the morphia exist in the liquor? The crystals would suggest that it was simply as a hydrate ; but if so, by what agency is it held in solution? A point of great interest, upon which I would like to have some evidence, is the stability of morphia under the treatment the opium undergoes in the preparation of the extract, or of the purified tincture recommended by these authors. They inform us that the crystals obtained from repeatedly evaporated extracts, differ considerably from those obtained from the same liquor before the repeated evaporations ; and we learn from them that certain colloid matters (*e.g.*, such as are contained in wine) detract from the crystallisable character of the alkaloids known to be present ; but we have yet to learn whether the change effected by repeated evaporations consisted of the separation of colloid matter in the form of brown deposit, which at each evaporation is separated as an insoluble residue, or whether the change is in the acids with which the alkaloids are combined, or even in the alkaloids themselves.

I am glad to note that the paper is given as a preliminary research, and the paper which followed the year after was a continuation, but not a completion, of this interesting subject, and I hope we shall be favoured with a large addition to these observations at some future time. I am not surprised that the writers experience a tendency in the task to expand as they progress ; I commenced the analysis of opium without any desire to make it a subject of study, but the great

complexity of the material, the conflicting results obtained by different analysts, the instability of some of the constituents, and the difficulty of separating others, soon seemed to open out an endless list of queries and of reactions to be tried. My experience has not to any considerable degree run parallel to theirs, but so far as it has done so it has confirmed their observations; apart from their microscopical results, they summarise their conclusions as follows :—

“ That tincture, most nearly of any of the preparations, represents the properties, good and bad, of the crude drug.

“ That when crude opium is taken up with proof spirit as tincture, the resin separates on evaporation.

“ That the preparations which have held their ground with the public and the medical profession, in spite of price, differ from tincture in their comparative freedom from resin and narcotine, and in containing only a diminished quantity of meconic acid.

“ That in the preparation of extract of opium it is important to use a large quantity of distilled water to insure the separation of narcotine and resin.

“ That when extract of opium is dissolved in water, filtered and evaporated again to an extract a second or third time, the crystals frequently differ considerably from those seen in the normal or first formed extract.

“ That when extract of opium is taken up with rectified spirit 56° over proof, and evaporated again to an extract, crystallisation does not take place, or only to a very trifling extent.”

They regard the narcotine and oleo-resin as the objectionable constituents, the morphia and codeia as those which act beneficially, and with regard to the bitter extractive, they say “ though opinions differ with regard to its precise properties, it seems at any rate to increase the narcotic power of the more active constituents.” And their second contribution concludes with instructions for the preparation of a purified tincture of opium, which contains these advantageous prin-

ciples, and excludes those which are objectionable. The process consists in exhausting opium with cold water, and evaporating to an extract; dissolving this again in a large bulk of cold water, the resin and narcotine, which were retained in solution while only a small portion of water was present, are now precipitated, and after subsidence are separated, the clear liquor being evaporated again to the consistence of treacle, and then mixed with a considerable proportion of spirit by which gummy matters are separated; the clear solution thus obtained being diluted with water till its bulk bears the same relation to the opium employed, as the bulk of the tincture which might have been obtained from the same quantity of the crude drug.

The resulting solution is pale brown, and free from the characteristic odour of opium, and appears to carry out the correct principles of pharmacy as applied to opium, except as regards the one point upon which I have laid most stress; that is, the quality of the opium from which it is prepared should be precisely defined, at least as far as regards the percentage of morphia it contains.

I have already alluded to the particulars which Mr Battley published many years ago, of the use he made of cold water as a solvent for the active matters of drugs generally, and perhaps we might say of opium in particular, though of this point he does not precisely inform us. His general rule is to extract with a small bulk of water, because the water, when used in about the proportion of two parts to one of the drug, has a considerable power of dissolving resinous and other matters together with the gum, &c., and to avoid any considerable evaporation, because it renders many of the active principles insoluble. These are just contrary to the rules commended by Messrs Dean and Brady for the extraction of opium; and I must add, just contrary to what we should suppose Mr Battley had used for the preparation of his liquor of opium, judging from the microscopical characters possessed by his liquor, and by the aqueous solutions prepared by Dean

and Brady's process, and by the contrast presented by the crystalline residue of Battley's liquor to that of tincture of opium, in which we know that the whole of the crystalline constituents are fully represented.

It is not unlikely that Mr Battley was guided by the principles he had arrived at, and exactly reversed the rule, seeing that in the case of opium he did not, as in most other drugs, wish to include all the active matters, but rather to include only one or two, and to exclude as many.

There is one point in which Mr Battley's rules may be considered superior to those of Messrs Dean and Brady, that is, that he obtains some degree of uniformity in the strength of the ultimate product, independently of the varying quality of the crude drug upon which he operates, his standard being to evaporate his infusions till a specific gravity of 1·2 is obtained, no matter what relation may subsist between the bulk of the product and the quantity of raw material required to produce this result; but as the density may result from inert or objectionable matter, in the case of opium, we can only conclude that he has either perfected his process so as to eliminate these materials, or that his liquor must vary in strength. Messrs Dean and Brady's observations would rather indicate that the former of these suppositions is correct, for they say, "We have examined it frequently, and always have met with the same characters." Even though this be the case, we can scarcely suppose he obtains results so uniform as would be obtained by standardising the opium, as I have suggested, to a precise percentage of morphia.

In conclusion, it will perhaps be well to indicate very briefly some of the difficulties which are met with, and which are to be overcome, in the process of estimating morphia in opium.

One important element in this operation is *time*. The influence of time is perhaps the greatest difficulty to be contended against by any one who has to work the analysis in the moments he can snatch from business between other engage-

ments. I have repeatedly operated upon a bulk, say 300 grains of opium, dividing the product into three parts at some particular stage of the process, that I might make a fair comparison of the advantages of three modes of treating the product at that point; but the time which elapsed between the various portions made so great a change in those which had been kept as to make the comparison of but small value. For example, working upon 300 grains by the official method up to the point where the lime decoction had been acidulated and evaporated to  $1\frac{1}{2}$  ounce, and divided into three portions, one for immediate use, and two kept in  $\frac{1}{2}$  ounce phials for subsequent examination. It was expected that the liquor was in a condition little liable to change by keeping; but on returning at another time to the contents of these phials, the great bulk of both morphia and colour had been deposited; and, though the former could be dissolved again by the application of heat, much of the colour remained insoluble, and the character of the liquor was so far changed as to vitiate any results as the basis of a comparison between the old and the new methods.

Again, in the use of bicarbonate of potass to precipitate colour and narcotine from the lime decoction after acidulation and evaporation, the time which is to be allowed for their deposition must be long enough for this purpose, and not long enough to risk the precipitation of morphia also. The time I have indicated as at least approximately satisfactory is half an hour, though I do not know that a longer time would not be desirable; a considerable portion of the lime remains in solution along with the bicarbonate of potass, and is afterwards precipitated as carbonate along with the morphia, unless the carbonic acid is got rid of by either time or heat, when the bicarbonate is decomposed by hydrochloric acid previous to the precipitation with ammonia. Then, as regards the time required for the morphia to precipitate, I have found a further deposit take place in the filtrate and washings, when the morphia had been allowed twenty hours to deposit. It



may be that the prescribed twenty-four hours even is too short, and that the temperature affects the time required.

Another difficulty that obstructs definite conclusions regarding the merits of different modifications of the process, is the great influence which the impurities or accompanying matters have upon the reactions of the alkaloids, &c., with such solvents or precipitants as may appear advantageous from a study of the behaviour of the proximate principles in the isolated state. We may ascertain to what extent hydrochlorates of morphia and narcotine are precipitated by lime or ammonia when dissolved in pure water or spirit, but we cannot safely conclude that the same results will be obtained in the presence of the gummy and extractive matters of the opium, nor can we feel sure that the results obtained with one sample of opium will hold good in the examination of another in which the proportion of crystalloids and colloids are different.

## LECTURE XXVIII.

### PHARMACY OF SPECIAL DRUGS.

#### ALOES.

ALOES may be said to have irritated the pharmaceutical rectum for the last thirty years.

We find in the "Pharmaceutical Journal" for 1842-3 a commencement of the discussion of the merits and demerits of compound decoction of aloes, which has gone on with more or less interruption from that time to this. The decoction has been many times commended as a valuable preparation,—a mode in which aloes could be given when it disagreed in the form of pills,—a preparation, from the administration of which we could obtain laxative action more resembling the natural action of the bowels than when exhibited in any other state. It has been said that in this form a larger dose could be tolerated, and that it was free from the tendency to griping which attaches to all the other modes of giving this drug; and it has been condemned as variable in appearance, variable in action, unscientific in principle, and only deserving of being expunged.

Regarding the drug itself, there is likewise a similar contradiction in respect to some particulars: the tendency which different varieties have in different degrees to cause alvine irritation, the consideration of which properties has led some authorities to prefer Socotrine aloes to Barbadoes, and the aqueous extract to either; has also led an authority, no less than Professor Christison, to give a preference to the Barbadoes aloes, and to say that only those descriptions of aloes inferior



to Socotrine were improved by the process of extraction. Some authorities lay great stress upon the superiority of the extract to the crude aloes, and even urge the superiority of the cold water extract to that in which heat has been used; while, on the other hand, Christison and others regard all varieties as much alike, excepting in degree of activity, stating that the irritation is proportional to the purgative action, and that the same effects may be obtained by any variety by regulating the dose, using a little less of Barbadoes, or a little more of Cape aloes, than would be used of Socotrine; and quite recently we have had aloin lauded as containing, in a considerably concentrated state, all the virtues without the vices of aloes; and, on the other hand, it has been stated to have little action, and that little depending upon the oxidation which it may undergo out of or in the system.

These incompatible statements indicate a very unsatisfactory condition of our knowledge of the pharmacy of aloes, and a necessity for careful experiments, in which the testing of medicinal activity should afford a constant comment upon the chemical or pharmaceutical products.

In treating of aloes, it was originally my intention to supplement the chemical and pharmaceutical experiments which I had tried, and the much more numerous and important experiments of Dr Tilden and others, with a few further experiments upon the activity of the various substances into which aloes may be separated. I commenced trying the effects upon myself of aloin, the aqueous extract, the resin, the aloes which had become insoluble from the action of air and water, the aloes which had become free from bitterness by the action of alkali, air, and moisture, the aloes which had become insoluble from the action of sulphuric acid, air, moisture, and heat; and I proposed to repeat these experiments, and to add to them observations upon the activity or otherwise of aloes which had become insoluble under the action of warmth, water, and phosphoric acid, and another portion of aloes which had lost its solubility under the action of sul-

phuric acid, air, and water, at a temperature varying between 60° Fahr. and 90° Fahr. But the condition of my health would not allow of my carrying out to indisputable results the course of experiments I had designed. I place so little confidence in the testimony obtained by using the materials upon a variety of patients, that I have not thought it worth the trouble of seeking to follow up the subject in a way which I could only regard as unsatisfactory ; I, therefore, concluded to leave the matter to the chances of the future, not doubting that in course of time there will be found some experimentalist with a robust constitution, or some one willing to be a martyr in the cause of science, of pharmacy, and of aloes ; but while I am not permitted to do as I had wished with my subject, there is yet ample room to review the problems which have engaged pharmaceutical attention in past years, and the progress that has been made towards a settlement of the question ; it may be also to note the pertinacity with which old ideas and old practices are retained, even in the light of what appears to be superior knowledge, and certainly to note the difficulty of coming to definite conclusions in relation to the activity of drugs, even in so palpable a case as regards the purgative property of aloes.

The circumstance which first strongly directed my attention to the activity of aloes and the changes which take place in it was an observation made in 1869, and to which I alluded in a former lecture, but which it will not be inappropriate to describe more fully now.

A specimen of aqueous extract of aloes, which had been kept some time, and had become darker than its usual colour, was examined with the view of ascertaining if, with the change of colour, it had also lost in any considerable degree its solubility, and presumably also its fitness for use. 100 grains were treated with warm distilled water and left 7 grains insoluble, besides which the warm solution let fall a further deposit on cooling.

The question naturally arose, What is the medicinal value

of this portion which has become insoluble, and has it become insoluble by the long keeping of the extract in the powdered condition, or was the insolubility the result of the evaporation to dryness of the solution of aloes? The statement of Battley, that extracts generally are injured by evaporation beyond the liquid condition, giving a little more force to these considerations.

The cold solution being filtered perfectly clear, was set to evaporate below 212° Fahr. When reduced to dryness it did not entirely redissolve in water. The evaporation was repeated twelve times in the course of a month, and at each time a portion was reduced to the insoluble condition; if the evaporation was not pushed to dryness, the deposit took place on cooling.

The matter thus spoken of as insoluble would probably be more correctly described as sparingly soluble, for the washings which were brown at first became paler up to a certain point; but as far as the washing was pursued they continued to be coloured, and the pale colour of the latter portions did not become noticeably paler as the washing was continued. This sediment was of a grey brown colour, and free from bitterness.

The precipitate on the filter was treated with solution of potash, in which it dissolved freely with the production of an intense brown colour, the solution being still free from bitterness, showing that the absence of taste was not simply the result of sparing solubility.

To the clear alkaline solution acetic acid was added, which caused the reprecipitation of the brown matter which was slightly soluble in excess of the acid. The portion not dissolved by the acid was further partially dissolved by treatment with spirit, and the portion left by spirit was totally and freely taken up by liquor of ammonia, with the production of a deep purple brown solution.

The original aloetic liquor from which this precipitate had been separated was continued under the same treatment of evaporation and solution, and for two months more continued

to deposit a sediment at each repetition of the process. The residue had necessarily become very small, and the deposits had diminished in even greater ratio, and it was considered unnecessary to push the process further. Of the original 100 gr., there then remained  $3\frac{1}{2}$  gr. of "highly purified" extract of aloes, which possessed but little bitterness, and in that dose caused no griping, nor any other effect upon the bowels.

In a subsequent experiment on 60 gr. of a new sample of extract of aloes, similar results were obtained, but the experiment was stopped when 10 gr. of precipitate had been obtained; the 10 gr. being made into a mass with soap and divided into three pills, of which one was found to act as a mild laxative.

The portion of aloes which remained soluble at this stage was tested in the same way, and 3 gr. made into a pill with soap proved a little more active than the pills made with the precipitate, though less active than an equal quantity of aloes which had not been subjected to this treatment. Perhaps I can best describe its activity by saying that 3 gr. were about as active as 4 gr. of the official pill of aloes and myrrh, which contains only  $1\frac{3}{4}$  gr. of aloes.

It seems not unlikely from these results, imperfect though they be, that aloes is not increased in activity by the conversion into an extract; but more of that presently.

The next experiment was with the view of obtaining the change in aloes which takes place in the official decoction, but not complicated by the presence of the other ingredients usually added.

Sixty gr. of extract of aloes rubbed with 30 gr. of carbonate of potass, and 10 oz. of distilled water, formed a clear solution, which was not quite so bright after boiling five minutes. Water being added to compensate for the loss by evaporation, it was divided into three equal parts A, B, and C.

C, mixed with  $1\frac{1}{2}$  oz. of proof spirit (to represent the tincture of cardamoms of the official decoction), looked a little brighter for the addition.

B, preserved without addition.

A, evaporated below  $212^{\circ}$  Fahr., became very dark brown, and almost lost its bitterness by the first evaporation; but after continued evaporation and solution for fourteen days, it still retained its solubility in water, but even then had not *entirely* lost the slight bitter smatch which it was found to possess at the end of the first evaporation. After the condition of the liquor had been examined, it was again evaporated to dryness and treated with rectified spirit, which was found to be almost without action upon it; it was therefore redissolved in water and evaporated to a syrupy condition, and poured into 2 oz. of rectified spirit; the bulk of it was precipitated, the liquor on evaporation leaving 10 gr. of brown deliquescent residue, having a *slight bitter* taste. The precipitate being washed through the filter with water and evaporated, left 20 gr. of a brown black, slightly hygroscopic residue, with an empyreumatic taste, but free from bitterness, the taste being principally that of the carbonate of potass present.

Five gr. of it taken as a pill produced no effect, a second dose of 10 gr. was then tried with no result; nor was there any appreciable action from a 5-grain dose of the residue of the alcoholic liquor.

I think we may safely infer that the activity of the aloes had departed along with its bitterness, and I anticipate I might have come to this conclusion with regard to the insoluble deposit from the evaporation of the simple aqueous solution of the extract of aloes, had I not been content with observing what I imagined was a mild laxative action, but which might have been an imaginary action. A repetition of the experiment, especially in larger doses, would have settled this point, had I not been compelled, as a matter of prudence, to desist at least for a time from experiments of this character.

The carbonate of potass in this experiment evidently greatly promoted the change in the aloes, for the bitterness had more completely gone by the first evaporation in this experiment than in three months in the case quoted above, and it became

a point of some interest to ascertain if the presence of an acid would prevent the decomposition of the aloes.

Two solutions were prepared, each by boiling 50 gr. of extract of aloes in half an oz. of water, and 2 drachms of diluted sulphuric acid, diluting with  $1\frac{1}{2}$  oz. of water, and filtering when cold, and submitting the clear liquors to evaporation at different temperatures, one being at heats varying up to about  $190^{\circ}$  Fahr., the other not exceeding  $90^{\circ}$  Fahr.

In the former of these, as might be expected, the aloes was soon converted into a carbonaceous mass, a few evaporations extending over a few days, being sufficient to destroy the bitterness, leaving a black residue weighing about half as much as the original extract and void of activity.

The other solution was repeatedly evaporated and dissolved during a month; it continued of an aloetic brown colour. A portion was precipitated by the first evaporation, and increased in bulk with each repetition, but at the end of the month there was still so much soluble as to impart to a few oz. of water a clear brown colour and bitter empyreumatic taste. No experiment was made of the medicinal value of either the soluble or insoluble portions; the acid in the liquor being neutralised with chalk, the solution retained its colour and its bitterness.

A similar solution of extract of aloes was prepared, substituting phosphoric acid for sulphuric; this acid being selected on account of its fixity and its freedom from corrosive action; the clear solution being exposed to heat varying up to  $190^{\circ}$  Fahr. After several evaporations, a portion of the extract separated as a soft clot, differing materially both from the cindery mass produced by sulphuric acid at this temperature, and from the powdery sediment deposited in the pure aloetic solution. The repeated treatment with water and evaporation being continued for a month, there was still a portion left soluble sufficient to impart a brown colour and slight bitterness to the water. The change in this case was less rapid than in that where sulphuric acid had been used, or where carbonate



of potash was present, but more rapid than where the extract was dissolved in pure water; but the activity of the insoluble part was not tested.

Reverting to the two samples of decoction of aloes which I mentioned before, the portion B, which was preserved without addition, appeared to have undergone no change by keeping about three months, except that it had become quite bright, and a *very small* deposit had taken place, the colour, odour, and taste being just the same as when new, judging by comparison with a new sample, prepared in exactly the same way; and the same may be said of that portion to which spirit had been added, the colour, odour, and taste were similar to a new sample containing the same addition of spirit. The new sample was not quite bright; the old was quite bright, and there was a very small sediment in the bottom of the bottle. Both samples had been kept corked, not exposed to strong light, and at a temperature ranging between 45° and 60° Fahr.

While making the new decoction for comparison with the old, I kept a small sample of the cold solution separate before boiling. It was found that the boiling had very much modified the taste; it was much less bitter, and the bitterness had less of the nauseous characteristic taste of aloes.

It appears probable from these observations that the aloes become modified by boiling with carbonate of potash, but not further changed by keeping if the air be excluded. I may mention in connection with this, that a bottle of wine of aloes (which is said to lose its bitterness by keeping) appears to have undergone no change during two years, the bottle being full, and the stopper rarely removed, it became turbid on frosty mornings, but bright again as it got warmer, and has the full bitter nauseous aloetic flavour.

I cannot say that I have ever seen a sample of decoction of aloes which had lost its bitter taste by keeping, and I could scarcely have supposed that any ordinary keeping would have permitted so much atmospheric action as I have found neces-

sary for the destruction of the bitterness in the samples I have purposely exposed. We must probably take the statements as approximately rather than strictly accurate ; but if we have to admit that the change effected during decoction is only the commencement of a series of changes which will go on more or less rapidly, according to the mode of keeping, and which would ultimately terminate in a total loss of activity, there appears good reason for looking upon decoction of aloes as an unsatisfactory preparation. And if we admit, as the testimony of some would seem to give grounds for, that there is a stage in the change at which the bitterness has disappeared, without a total loss of activity, and at which point they attribute to the aloes some valuable advantages, we are still in a most unsatisfactory position, being unable to determine by the taste or other physical qualities whether our preparation is at this stage, or has passed beyond it to one of utter worthlessness.

If there is a condition of aloes in which its action is sublime, it surely is a pity if we cannot perceive that further change, which converts it from the sublime to the ridiculous.

With the view of testing the value of aloin and the resin of aloes, I operated upon one pound of Barbadoes aloes according to the process described by Dr Tildin at the Pharmaceutical Conference, 1870.

The pound of aloes, not of a carefully selected quality, but by commercial designation, "good seconds," was dissolved in 1 gallon of boiling water, acidulated with  $\frac{1}{2}$  ounce of diluted sulphuric acid ; it dissolved freely, forming a nearly clear solution, which on standing 14 hours, had deposited a soft resinous mass, which was separated, dried, and weighed  $3\frac{1}{4}$  oz.

The clear liquor evaporated at about  $160^{\circ}$  Fahr. to 32 oz., and set aside to crystallise, showed no change during the first 24 hours,—in three days a considerable yellow crust had deposited, which, when collected, strongly pressed, and dried, weighed 1 oz., and was pale yellow.

The mother liquor, during the next three days, deposited aloin in a loose condition, which, when finished, weighed  $1\frac{1}{8}$  oz.



The mother liquor being allowed other three days, let fall a small crop amounting to  $\frac{1}{8}$  oz., making altogether  $2\frac{1}{4}$  oz. of aloin obtained from 1 lb. of aloes; how much aloin was contained in the aloes, or how much might be produced from it, are distinct questions. I simply give the product I obtained.

The mother liquor from the last crop was neutralised with 50 grains of carbonate of lime, filtered and evaporated to dryness, it yielded 7 oz. of extract. The rough analysis stands thus—

Resin . . . . .	3·25
Aloin . . . . .	2·25
Extract . . . . .	7·00
Loss and moisture . . . . .	3·50
	<hr/>
	16·00

The loss being principally the liquor absorbed by the strainers and the cloths in which the aloin had been pressed, but includes a portion of water contained in the aloes, which was far from dry at the commencement. I commenced taking a few doses of the resin, which acted with rather less energy, and, I believe, a little more pain than an equal dose of aloes.

A few doses of the extract were next tried, and were found rather more active than the resin, probably about the same as aloes in its normal state.

I began trying doses of the aloin carefully, for fear of greater activity, though I could not see that there was any great activity to be accounted for, considering that of the  $12\frac{3}{4}$  oz. of product, 7 oz. were freely active, and 3 were only a little less so. After several trials, I concluded that the aloin was somewhat more active than the extract. But I must again repeat that my experiments upon this point were cut short without having resulted in conclusions so definite as I had hoped. My impression was that  $\frac{3}{4}$  of a grain to 1 grain of aloin produced effects comparable to 4 grains of pill of aloes

and myrrh, a standard with the action of which I was familiar, and which contains  $1\frac{1}{2}$  grain of Socotrine aloes. It had been my intention to ascertain how much of each of the preparations was required to produce pills of a strength equal to these, and the trials I have recorded were regarded as merely preliminary to a more careful series, which I may probably never perform.

From a general view of my experience, I am disposed to think that the extract is not more active than the entire aloes; there is evidently activity lost when the evaporations are repeated, and no doubt there is some lost at each evaporation, probably as much during the first evaporation as would counterbalance the increased activity resulting from the separation of the deposit, which has a purgative action below the average of the entire mass. Any superiority, if such exists, in the extract, must be looked for in a diminution of irritant qualities, resulting from the removal of the resin.

In the "Pharmaceutical Journal" for September 1842, the editor, commenting upon some suggestions for the improvement of decoction of aloes, says that a large portion of the aloes is soluble in solution of carbonate of potash, without heat; that heat effects a change in the soluble constituents, "which are thus rendered to a certain extent insoluble;" and he concludes with the suggestion that an infusion should be adopted instead of a decoction.

A few months later, a similar recommendation is made by Mr Fisher.

In November 1846, Mr Westall makes suggestions for improvements which chiefly consist in making it double the strength, so as to be more alcoholic, and to represent the official strength when diluted with its own bulk of water. In the British Pharmacopœia, the aqueous extract of aloes was substituted for the crude drug in the preparation of the decoction; some of the former criticisms consequently do not apply to the preparation as now made.

In the "Pharmaceutical Journal" for August 1868, we find an

article on aloes quoted from "Medical Times and Gazette," in which the writer, after quoting Mr Squire's statements regarding the relative value of Barbadoes and Socotrine aloes, as stated in Squire's "Companion to the Pharmacopœia," says "aloes should always be given in the solid form. It takes twice or thrice the quantity in solution to produce the same effect. All the tinctures, wines, and elixirs are as wasteful as nasty. Even the favourite decoction is a mistake. It were far better to give one grain of watery extract of aloes in a pill, and a dose of potash and cardamoms in a draught, than to waste four grains of extract in an ounce of the decoction."

And turning to a recent edition of Squire's "Companion" (1869), we read under the head of decoction of aloes—"It is a most valuable aperient, 1 oz. or  $1\frac{1}{2}$  oz. equal to 6 grains. Aloes act naturally without griping, whereas three grains of aloes in a pill would probably purge and gripe too. A valuable paper on aloes ("Medical Times and Gazette," January 4, 1868), records the fact that a very much larger dose of aloes can be given in solution than in the solid form."

I think we may now dismiss as settled the questions which were debated regarding the precipitation of aloes in the decoction by keeping, but I have as yet obtained no evidence upon which to determine the propriety of substituting an infusion for a decoction. It is most likely that time would effect by degrees the change which boiling produces promptly, and that by substituting an infusion we should have still greater variation between old samples and new. And if this were not the case, we should probably lose the only advantage, be it imaginary or real, which the decoction possesses over other preparations of this drug. These, however, are only speculations, and we must admit that the advantages of an infusion of aloes remains one of the problems which must be settled by a further study of the pharmacy of aloes.

Careful personal experience is probably the only way by

which to correct or to reconcile the discordant statements of the "Medical Times" and Squire's "Companion."

Supposing it to be admitted that aloes in solution in pure water, and exposed to the air, gradually changes into a less soluble and less active form; and that in solution in alkaline liquors a similar change takes place more rapidly, though without precipitation in consequence of the changed aloes being soluble in alkaline liquors, these considerations suggest a series of questions relative to the permanence of various other preparations. Does the aloes alter materially in drying and grinding? Is the powder liable to become oxidised and less active by keeping? Does its degree of dryness materially affect its permanence in this respect? Do aloetic pills change in like manner; and if so, is the change expedited by the presence of soap or other bodies having an alkaline reaction?

I have not collected any conclusive evidence upon any of these points, but I think we may consider the pills with soap liable to a slow change affecting the outside, but probably not sufficient to give a noticeable diminution of activity in ordinary keeping within a few years. The only evidence of which change that I can offer you is the following:—

A lump of pill of Socotrine aloes having been kept in a jar for eight months, the exterior was a little darker than the middle of the lump, and was paired off thinly. Five grains of these parings, dissolved in two drachms of water, gave a solution rather darker and less bitter than a similar solution prepared from the middle of the lump, but yet so bitter as to preclude any idea of its having lost its purgative qualities.

If it were desirable to do so, probably the addition of a little caustic alkali to the pill would expedite the conversion of the aloes into that modification, of which we might enjoy the privilege of taking six grains without experiencing a sharp catharsis.

The stability of aloes under the action of strong sulphuric acid is much greater than that usually possessed by organic substances of so complex a nature. You will remember the

pills I noticed in a former lecture, consisting of aloes made into a mass with oil of vitriol, which are said to act well, though, from their hygroscopic quality, they are inconvenient to keep.

Probably the addition of a little sulphuric acid to the solution of aloes in the aloin process may add to its stability, and if an aqueous solution be required in pharmacy, an acidulous one would probably be less liable to change than an alkaline liquor; but the neutral solutions appear to me to possess a sufficient degree of permanence if the air be moderately well excluded, and I could not advocate the addition to our preparations of an acid infusion of aloes, unless it was proved by further experience that the conjunction with acid was therapeutically advantageous.

The chemistry of aloes has received several valuable contributions within a few years, the most important of which are the papers by Dr Tilden, read at the meetings of the Pharmaceutical Conference, 1870 and 1872.

In various points, his statements agree with my experience; in one or two I am not prepared to agree with him, but without giving a critical examination of his results, I will simply subjoin a summary of them.

Aloetin, or dehydrated aloin, constitutes the bulk of all aloes, and is capable of becoming aloin in the presence of water.

Aloin, in its hydrated state, is crystalline, and is the body to which especially aloes owes its bitterness. Pure aloin may be repeatedly dissolved and evaporated to dryness with very little change. It dissolves in oil of vitriol without change, even when gently heated.

Resin of aloes is not a true resin, being partially soluble in hot water, and probably converted into aloetin by the process. Like aloetin, it yields chrysammic acid by treatment with nitric acid.

Aloin is rapidly oxidised in the presence of alkalies, air, and water.

Extract of aloes in solution with carbonate of potash

oxidises rapidly, and loses its bitter and purgative properties. The active constituent of aloes is still not definitely known, but is probably the soluble brown uncrystallisable extract.

In the latter contribution on this subject, Dr Tilden gives us the constitution of aloes,—

1. Aloin and products of its decomposition or change.
2. Resinoid matter.
3. Gum, albumen, salts, &c.

The relative activity of aloes and its constituents, especially aloin, had been the subject of a discussion at the intervening Conference, and an activity was attributed to the latter by several who are presumably in a good position to form a fair estimate, much greater than my personal experience would enable me to endorse, and greater than we could admit, taking into account the activity of the other constituents, unless we suppose that the total activity of the constituents of the aloes is increased by their treatment in the process of separation.

Another interesting article had also appeared in the interval from the pen of Professor Fluckiger, in which he shows that varieties of aloin, possessing marked characteristics, are derived from the different varieties of aloes. These studies are to be regarded as chemical rather than pharmaceutical investigations, and involve a refinement of operation greater than most pharmacists can apply; but the pharmacist's study will be greatly facilitated by the light shed upon the subject by these researches, and he will promote in the greatest degree the progress of his own department by adopting, wherever practicable, the philosophical habit of expressing his knowledge in simple and concise statements, expressed in the clearest language, and founded on the most unequivocal data.

## LECTURE XXIX.

### PHARMACY OF SPECIAL DRUGS.

#### IRON.

IN studying the pharmacy of iron, we have to deal with circumstances in many respects the converse of those which have claimed our attention in relation to the drugs which have been the themes of the last three lectures. Then we had to separate inert or objectionable matter, now we are dealing with a simple body. We have nothing to separate, but have to consider the states of combination into which it may be brought with the greatest advantage. In both cases we have to contend with a public disposition to multiply the number of preparations in use, the change frequently being a change only, and not an improvement.

The axioms which I proposed for the practice of vegetable pharmacy may be modified to some extent for application to our present subject.

The first requirement must be the production of such iron compounds as admit of being administered in mixture with any other drug that may be necessarily given at the same time. It is desirable to have preparations of iron suitable for giving along with acids, others for union with alkaline salts. At other times it may be desirable to give iron with iodide of potassium, or arsenical solutions; to use it in combination with astringent matters, or to have the iron-preparation itself as free from astringent properties as possible; and in all these varying circumstances something may be done towards getting satisfactory results, by studying and classifying the iron com-



pounds and their reactions. There appears to be a great deal of speculation, groundless speculation I believe I might say, regarding the advantages of iron in one form or other. The iron probably acts much in the same manner, and probably much in the same degree, in whatever form it be given, provided it is soluble; the difference observed depending upon the action of the acid or other body which imparts solubility, and the other substances which may be administered at the same time. The stomach is a great leveller; citrates, lactates, malates, or tartarates alike yield to its action; and the iron in combination with them probably acts just the same, whatever the organic compound may be.

In the year 1842 Professor Christison, writing of iron and its numerous preparations, said—"It is much to be desired that accurate inquiries were made as to their respective qualities. That the official preparations are unnecessarily numerous, no one can doubt who looks to the extreme simplicity of the action of chalybeate remedies. A considerable difference will probably be found to exist between those where the iron is in the form of protoxide, and those in which it is present as a sesquioxide; and the former are generally, and perhaps correctly, considered the more active."

There is no article in our *materia medica* which has been administered in so many different forms as iron, and none which threatens to go on multiplying and replenishing our shelves with such frightful fecundity.

To stay this plague, to increase the death-rate, and diminish the birth-rate of iron remedies, is the one thing most highly to be desired in connection with our present subject. Before considering the means by which this is most likely to be accomplished, we must give a little consideration to the official preparations of iron, and to the more important of those which are non-official.

Iron itself may be obtained in a state of almost absolute purity in the form of wire, the soft wire used by wireworkers being found to contain 99.9 per cent. of pure iron.



The bulk of the iron used in pharmacy, however, is not obtained first in the metallic state, but from the decomposition of sulphate of iron, which in its turn was produced by the oxidation of iron pyrites. Though the crude sulphate of iron of commerce is not by any means a pure salt, the amount of contaminating metals is insignificant; traces of zinc, copper, or aluminium being the most prevalent; but the presence of persulphate is always to be detected, and usually in notable quantity.

The iron pharmaceuticals may be said to be liable to accidental impurity, but not to adulteration, the cheapness of the raw material being a protection against the temptation to wilful admixture, but affording no safety against carelessness of manipulation. The official processes generally provide satisfactory precautions, but at more than necessary cost of labour or material, and manufacturers frequently obtain equally good results by other means. This is the case with the granular sulphate of iron, which is sold at prices which preclude its being made from iron wire and sulphuric acid; but as I showed you in my lecture on Crystallisation, it is satisfactorily pure even when thrown down from solutions of the commercial sulphate of iron containing a free admixture of the peroxidised salt; and it matters not whether it is prepared by one method or the other, so long as the point aimed at is accomplished. The granular sulphate of iron may be said to be cheap, pure, and permanent. Perchloride of iron in solution is not readily prepared of an equal degree of purity by the official process, or by the older processes which it has supplanted; but the impurities which are present either in that made according to the official formula or by dissolving hydrated peroxide of iron in hydrochloric acid, are not of such kind or quantity as to affect its medicinal value, unless it is contaminated with unusual impurities, or is deficient in strength through carelessness with regard to the quality of the chemicals used; but if so, this is the fault of the operator, and not of the process.

In the article as purchased, a little excess of hydrochloric acid is usual, and is desirable. It is also commonly impregnated with nitrous gases, which, though they are evident enough in the concentrated liquor, are not enough to affect the diluted solution or the tincture.

The next most important iron compound is the citrate with ammonia. It is not a definite salt; but there is no difficulty in defining the proportion of its constituents which is considered desirable, nor in producing it of constant composition. The commercial article is not subject to any serious variation, and its quality is easily ascertained by a series of readily applied tests.

These three preparations may be considered to stand apart from all the other preparations of iron in respect to their superiority, when we view them in all aspects, their well established therapeutic value, the ease and certainty of their preparations, their permanence, and the readiness with which they meet the requirements of almost every combination in which it is desirable to administer iron—their perfect solubility, and consequent ready absorption, and uniform action.

Peroxide of iron, like the three preceding, is readily and cheaply obtained of good and uniform quality; but however good and uniform its composition may be, its action is not so reliable as that of the soluble compounds previously spoken of. The condition of the stomach of the patient will, in all probability, materially influence the activity of preparations of this class, which are only dissolved in the presence of acid. It has sometimes been administered in extravagant doses; but the effects obtained are not proportionate to the quantity given, but to the quantity which is absorbed into the circulation, and this in turn will be determined more by the quantity of acid in the stomach than by the bulk of the material taken; but it will be determined also to some extent by the physical condition of the oxide. If it is overheated and rendered anhydrous, it is almost inert.

The action of iron upon the system, being essentially slow

in its nature, we cannot attach any value to the gradual solution and continuous action, which might have been desiderata had the action of the soluble compounds been rapid and evanescent.

The magnetic oxide is readily prepared of definite composition, and is not notably a changeable article; but there does not appear any grounds for expecting any advantage, therapeutic or otherwise, in its use.

Saccharated carbonate of iron is not of certain composition. Theoretically it might be expected to be uniform; but though the materials used are so cheap as to put it beyond any temptation to prepare it otherwise than correctly, it is reported by those who have taken samples from actual use, to be very variable. It probably does not possess any therapeutic superiority to compensate for this serious defect.

Mixtura Ferri Composita may be regarded as superior to the saccharated carbonate of iron in constancy of composition, *if prepared at the time it is dispensed*, because in that case its quality as sent out is dependant only upon the quality of the sulphate of iron and carbonate of potash used in its preparation; but its rapid change of colour, though it may not be accompanied with an equal change in medicinal value, is a trouble to the pharmacist, and a source of uneasiness to the patient from the impression which it is apt to leave, that the medicine spoils by keeping, or that the new supply is not the same as the old; but these little inconveniences are as nothing compared with its superiority in affording a definite dose of almost pure carbonate of iron in suitable condition for taking. If carbonate of iron is therapeutically demanded, this appears to be the best form yet adopted for its administration.

Iodide of iron, including the pill and the syrup, may be classed among the unsatisfactory preparations which have given trouble to pharmacists for many years past, and do not appear to have accomplished anything commensurate with the popularity they once possessed. They were introduced upon theoretical grounds. The idea of combining the tonic action

of iron with the alterative properties of an iodide appeared a happy thought ; but experience does not seem to have justified the great expectations, and the fashion which prevailed only a few years ago is now yielding before the newer fashion of giving iron syrups in which phosphoric or lactic acids are supposed to develop new virtues in the old base.

The phosphate of iron is, to some extent, a variable salt. The degree of oxidation it possesses depending in some measure upon the exposure to the air in the process of washing and drying. It is not much used except in the form of syrup, in which state it is not free from liability to further change by keeping.

Reduced iron is essentially a variable compound, both as regards its officially recognised composition, and in a still greater degree its commercial quality. I do not know any grounds, chemical or physiological, upon which it can be expected to possess advantages over the oxides of iron. It is not more agreeable to take than the latter bodies, and is less agreeable in effects, in consequence of the unpleasant eructations which it produces.

Tartarated iron may be dismissed with the simple statement that it does not appear to have any advantages which the citrate with ammonia does not possess in at least an equal degree.

The tincture of acetate of iron is very unsatisfactory. The great bulk of the iron separates when the tincture is long kept, a decomposition which has been more troublesome with the neutral acetate as now produced by double decomposition, than was the case in the old method of making a similar tincture by macerating peroxide in acetic acid, in which latter case the acid was usually in considerable excess.

Liquor of perntrate of iron is liable to the same species of deterioration as the tincture of the acetate ; the liquor of the persulphate, on the other hand, is a permanent and satisfactory trade chemical, though scarcely itself a pharmaceutical

preparation of iron, its use being confined to the manufacture of other iron compounds.

Wine of iron has nothing to recommend it but the taste. It is not very active, is very indefinite, and not tempting in appearance. The wine of the citrate has at least the advantage of greater uniformity.

Aromatic iron mixture may be said to have all the advantages of the *vinum ferri* without its pleasant taste.

Looking over the long list of iron preparations which have been introduced into practice, on the authority of some influential name, and in many cases also made official by one or other of the British or foreign Pharmacopœias, we fail to detect any general principle which has guided their introduction, or any common defect which characterises those which have enjoyed but a short-lived popularity. Comparing those which have been discarded with those which have supplanted them in the official list, the change is commonly for the better as regards uniformity of quality or permanence; but this cannot be said to be characteristic of the many new claimants for popularity, which have not been, and I hope will not be, admitted into the Pharmacopœia.

Among the rejected compounds we have ammonio chloride of iron, which has naturally fallen out of use as having no advantage over the uncombined perchloride. The indefinite mixture of carbonate and oxides formerly in all the Pharmacopœias under the name of carbonate of iron, and the iron rust of the Edinburgh College, have both been displaced by the more uniform preparation of pure hydrated peroxide of iron of the British Pharmacopœia. The solution of oxysulphate has fallen out of the list before the liquor of the pernitrate, not because it was more worthy of rejection, but probably because the latter solution was the hobby of a better rider. If so, may he soon ride it to death!

The "*Tinct. Ferri Pomati*" and the "*Tinct. Ferri Cydoniati*" were probably better because more active and more uniform than our "*Vinum Ferri*," though the juice of quinces or of

crab apples cannot be regarded as of sufficiently uniform solvent power to insure a good product. And the "Tinct. Ferri Aurantiaca" which, like the preceding, was official on the Continent, depended for its efficacy upon the solvent action which Seville orange juice exerts upon iron filings. It would be well if our vinum ferri would follow them into oblivion.

If we turn to the list of new iron preparations which we might hope to find based upon more scientific principles, there is discovered just the same lack of system and judgment. Judging of the future by the past, many of the preparations of to-day and of to-morrow will, in twenty years' time, be among the obsolete remedies, which have not improved the practice of medicine, nor in any way promoted its progress, unless by illustrating the folly of fashion. Many of them give trouble at their birth, and some continue to do so till they have passed away.

The granular effervescing preparations known as citrate of iron, citrate of iron and quinine, carbonate of iron, and iodide of iron, form a little family. It were better they had never been born. Their birth showed but the folly of their parents, and their decay shows that they did not possess the stamina for a useful existence.

Iodate of iron, if ever it be successfully introduced, will no doubt owe that measure of success to a repetition of the expectations which formed the basis of at least a temporary popularity for the iodide; it is said to be definite, stable, and worthy of further examination.

Malate, lactate, and the syrup of lacto-phosphate have all been recently advocated; it may be that they are all improvements upon some unsatisfactory articles which have preceded them, and perhaps that is the best we can say for them. The syrup commenced its career by showing a disposition to deposit its active matter by keeping; and, though it may be a triumph of pharmaceutical art to succeed in overcoming that difficulty, it would be a much greater triumph, taking a comprehensive view of the subject, to attain equally good



effects with the old, the well-known, the definite and stable compounds of iron, and that triumph, I have little hesitation in saying, was achieved before the syrup of the lacto-phosphate of iron was thought of.

The host of ferrated elixirs which have found a place in the so-called elegant pharmacy of America look too much like playing with physic to be made the subject of serious criticism. I should feel it a degradation to the British Pharmacopœia to be made the receptacle of such formulæ, a degradation from which I trust it will be saved.

The dialysed oxide of iron, and iron sugar, which contain iron in the state of a soluble modification of its peroxide, free from any acid or acidulous radical, will probably find at least a temporary favour with the French, sugar being the basis of their most approved remedies; but we have as yet no ground for supposing that they are likely to have any physiological action not possessed in an equal degree by the citrate of iron and ammonia.

I have now said quite enough against what I consider the unnecessary and disadvantageous multiplication of preparations of iron, and my next object must be to show in what way a few of those which have acquired a lasting reputation may in practice be combined with other drugs without involving deterioration or any objectionable decomposition, and, at the same time, allowing all the latitude which a prescriber may desire in the association of other therapeutic actions with the tonic effect of iron. This will necessarily include some consideration of what are commonly denominated the incompatibles of the iron salts. We every now and then hear mixtures spoken of as unchemical when there is no just ground for that imputation. It does not follow that a mixture is unchemical, or that its ingredients are incompatible when a decomposition takes place amongst them, unless the decomposition has been overlooked by the prescriber, and results in a detraction from the efficacy of the mixture, or the development of some unwished for property. Some of the

text books say that vegetable astringents are incompatible with the iron salts, no doubt because they produce with them a black, unpleasant-looking precipitate; but it may be that the inky-looking mixture is just as valuable as if no change of appearance had taken place; the tannate of iron being advocated by Benedetti as a chalybeate especially suited to chlorosis. The alkaline carbonates are also enumerated among the incompatibles of the sulphate and perchloride of iron, but the compound iron mixture of the Pharmacopœia is strictly chemical, and contains these so-called incompatibles; and if we find tincture of iron and aromatic spirit of ammonia prescribed together, it could only be regarded as unsatisfactory if the decomposition had been overlooked by the prescriber, a point which it is often difficult for the dispenser to determine; the only cases in which it is desirable for the dispenser to question the correctness of the prescription are where one ingredient unmistakably defeats the object of another, or develops some source of danger. I have seen syrup of iodide of iron prescribed in conjunction with an alkaline carbonate, and, though I had no hesitation in concluding that it was an oversight, I also had no hesitation in dispensing the mixture as it was prescribed; the action would not be dangerous, and, indeed, would probably be much the same as if no decomposition had taken place; but when we find perchloride of iron prescribed together with iodide of potassium, the case is more serious; a decomposition ensues, liberating iodine, the effect of which, even in small doses, is not to be trifled with. Keeping these circumstances before us, it appears to be the duty of the pharmacist to dispense as prescribed all mixtures in which no unpleasant consequences are likely to result from the decomposition, but to be prepared at any moment when the opportunity occurs of showing by what little modifications the unsightly, unpalatable, or inconvenient condition, resulting from decomposition may be avoided without materially affecting the general character of the mixture.



We will first suppose our task is to combine an iron salt with a vegetable astringent in solution free from the inky appearance which under these circumstances is usually produced. We must be guided by two considerations; the black precipitate is much less freely produced in the presence of free acid, or in the absence of peroxidised compounds of iron, and to illustrate the application of these facts I have here a series of mixtures—

No. 1.					
R	Ferri sulph.	.	.	.	} āā } gr. x. } ℥iv.
	Acid tannic.	.	.	.	
	Aquæ	.	.	.	
M.					

Divide into four phials, A, B, C, and D.

A has received no addition, and you perceive the usual inky deposit.

B contains 10 drops of dilute sulphuric acid, which has redissolved the precipitate, leaving the solution almost colourless and clear, in consequence of the precipitation not taking place in the presence of a small excess of a powerful acid.

C has a similar condition imparted by the addition of 10 drops of sulphurous acid, which acts as a reducing agent; the sulphate, which is entirely free from peroxide, yielding no precipitate with tannic acid, as may be shown by the power of sulphuretted hydrogen or metallic iron to produce a similar decolorisation. The addition of an alkaline salt to a solution thus decolorised causes a dirty pink precipitate; on exposure to the air, by which oxidation takes place, causes a purple black coloration; consequently, this is not so satisfactory a mode of preventing the colour as the use of a little free sulphuric acid, an addition which would rarely be objectionable when iron and astringent remedies were required.

D contains three grains of sulphite of soda as an addition, which has increased the black or pinkish black precipitate, in consequence of the soda taking the sulphuric acid and leaving the iron in combination with a feebler acid, not

capable of withstanding the precipitating power of the tannin.

## No. 2.

R	Tinct. ferri perchlor.	.	.	.	℥ss.
	Acid tannic.	.	.	.	gr. x.
	Aquæ ad.	.	.	.	℥ij.

M.

Divide into two phials, A and B.

A, having received no addition, contains a black precipitate, the clear portion also being purple-black.

B, which has been rendered freely acid with half a drachm of diluted hydrochloric acid, is olive green, and contains no deposit.

From these reactions I think we may conclude that, if it is *medically* desirable to administer iron with vegetable astringents, they may be regarded as chemically and pharmaceutically compatible in the presence of a little free mineral acid.

In actual practice we more frequently find that the astringent matter is incidental rather than an essential constituent of the mixture, and is present in comparatively small proportion. Infusion of gentian or of roses, tincture of cardamoms or of orange peel, being among the most frequent sources of the astringent principle, the blackening which takes place in consequence of their presence is, under any circumstances, less offensive than in the neutral mixtures described above.

The series of phials marked 2 C, 2 D, 2 E, 2 F, show the behaviour of compound tincture of cardamoms in such mixtures.

## No. 2 C.

R	Ferri sulph.	.	.	.	gr. v.
	Tr. card. co.	.	.	.	℥ij.
	Aq. ad.	.	.	.	℥ij.

M.

No. 2 D. The same, with the addition of 20 minims of diluted sulphuric acid.

## No. 2 E.

R	Tinct. ferri perchlor.	.	.	.	℥ <sub>xx</sub> .
	Tinct. cardam. co.	.	.	.	ʒij.
	Aq. ad.	.	.	.	ʒij.

M.

No. 2 F. The same, with the addition of 20 minims of diluted hydrochloric acid.

They show that the addition of acid materially improves the appearance, especially in the case of the proto-salt. While 2 C is the most objectionable, 2 D is the most passable of the four. With the perchloride, the colour, which is at first greenish black, becomes brown in the course of a day or two.

The next requirement is to provide suitable forms for the administration of iron in company with alkalies or alkaline salts. In the chemical handbooks it is stated that alkalies fail to precipitate iron in the presence of fixed organic substances, such as sugar or tartaric acid. This indicates the principle upon which our present object is to be attained, but it is necessary in the first place to note that in medicine and pharmacy carbonates of soda or potash are commonly spoken of as alkalies, but in chemical language the term alkali is restricted to the hydrates uncombined with carbonic or any other acid; and also to note that the completeness of the action must of necessity depend upon the proportion which subsists between the three substances—the iron salt, the organic matter, and the alkali.

With a given quantity of iron salt, one proportion of sugar will completely prevent its precipitation, while a smaller proportion will only prevent its being completely precipitated.

When it is desired to prescribe an iron salt with an alkaline carbonate, the carbonate of iron in the form of compound iron mixture, and the citrate of iron and ammonia, are quite appropriate, and probably leave nothing to be desired; but as we meet with cases such as those previously alluded to, we may advantageously give them a passing comment.

In the prescription where tincture of perchloride of iron

was ordered along with aromatic spirit of ammonia, a comparatively elegant mixture might have been produced if a little syrup had been added; thus—

No. 3.				
R	Tinct. ferri perchlor.	.	.	ȳss.
	Syr. simpl.	.	.	ȳij.
	Aquæ	.	.	ȳx.
	Misce et adde.			
	Spir. ammon. ar.	.	.	ȳss.

This, you will observe by the sample on the table, retains most of the iron in solution. It would have retained *all*, if ammonia and not its carbonate had been added; and it would have retained *none*, or next to none, if the aromatic spirit of ammonia had been mixed with the tincture of iron before the addition of the syrup, as is exemplified in the bottle marked No. 3 B, which contains the same ingredients mixed in a different order. In this case almost the whole of the iron has gone down as peroxide, the supernatant liquor being colourless.

In the bottle marked No. 4, there is the following mixture—

No. 4.				
R	Tinct. ferri perchlor.	.	.	ȳss.
	Syr. simpl.	.	.	ȳij.
	Aqua ad.	.	.	ȳij.
	Potass. bicarb.	.	.	ȳss.
M.				

And in No. 4 B, there is the same mixture, substituting 3 drachms of solution of potash for the 30 grains of bicarbonate. In this case you will observe the sugar retains the iron in solution in the presence of caustic alkali, but not in the presence of the bicarbonate.

No. 4 C.				
R	Sodæ tartaratæ	.	.	ȳij.
	Aquæ	.	.	ȳiss.
	Solve et adde.			
	Tinct. ferri perchlor.	.	.	ȳss.
	Sodæ bicarb.	.	.	ȳij.
M.				

The first effect of adding the tincture of iron to the solution of Rochelle salt is to produce a precipitate, which redissolves after the subsidence of the effervescence which takes place on the addition of the bicarbonate; the clear solution is then in a condition suitable for giving with lemon juice as an effervescing tonic.

No. 4 D is a form of saline tonic which we have frequently dispensed, and in which a decomposition takes place not accompanied with precipitation.

## No. 4 D.

R	Tinct. ferri perchlor.	.	.	.	℥ xv.
	Liq. ammon. acet.	.	.	.	ʒij.
	Aq. ad.	.	.	.	ʒij.

M.

The perchloride of iron is converted into peracetate, as is evident from the rich deep colour developed; the diuretic action of the acetate of ammonia is diminished by the conversion of a portion of it into chloride of ammonia.

In No. 5 we illustrate the behaviour of the proto-salt under similar circumstances, the mixture being as follows:—

## No. 5.

R	Ferri sulph.	.	.	.	gr. viij.
	Syr. simpl.	.	.	.	ʒss.
	Aquæ	.	.	.	ʒss.
	Potass bicarb.	.	.	.	gr. xx.

M.

And in No. 5 B the same ingredients occur with two drachms of solution of potash in the place of twenty grains of bicarbonate.

The former is an unpleasant-looking puddle, the latter a dark green solution almost free from sediment; it gradually grows darker by oxidation, eventually becoming a clear dark brown liquor.

## No. 5 C.

R	Syr. ferri iodid.	.	.	.	ʒj.
	Aquæ	.	.	.	ʒvss.
	Liq. potassæ	.	.	.	ʒjss.

M.

This is designed to show that the mixture, which I before described as having an unpleasant appearance from bicarbonate of potash being ordered along with syrup of iodide of iron, might have claimed a place amongst the preparations of elegant pharmacy by the simple substitution of an equivalent of caustic potash in place of the bicarbonate. It thus forms a dark green mixture free from sediment, and rather pretty in appearance.

It is perhaps self-evident that, when iron is to be used with oxidising agents, it should be in the peroxidised condition. The four following formulæ illustrate that proposition:—

No. 6.					
R	Tinct. ferri perchlor.	.	.	.	℥ss.
	Potassæ chloratis.	.	.	.	℥ss.
	Aquæ	.	.	.	℥ij.
M.					
No. 7.					
R	Ferri sulph.	.	.	.	gr. x.
	Potassæ chloratis.	.	.	.	℥ss.
	Aquæ	.	.	.	℥ij.
M.					
No. 8.					
R	Ferri sulph.	.	.	.	gr. x.
	Acid. nitr. hydrochlor. dil.	.	.	.	℥j.
	Aquæ	.	.	.	℥j.
M.					
No. 9.					
R	Ferri sulph.	.	.	.	gr. x.
	Acid. nitr. dil.	.	.	.	℥j.
	Aquæ	.	.	.	℥j.
M.					

In No. 6 there is no evident change, possibly there may be a partial decomposition, but not a total interchange of acids and bases, for when a hot saturated solution of chlorate of potash is added to an equivalent of perchloride of iron, the liquor on cooling deposits chlorate of potash free from iron.

No. 7 undergoes a gradual decomposition, the bulk of the iron being deposited as a basic chlorate of the peroxide.

In Nos. 8 and 9 there is necessarily no iron deposited,

though the salt is gradually peroxidised by the nitric acid without access of air.

Nos. 10, 11, and 12 illustrate the dispensing of iron along with iodide or bromide of potassium.

No. 10.				
R	Tinct. ferri perchlor.	.	.	3j.
	Potassii iodidi	.	.	3ss.
	Aquæ ad.	.	.	3j.

M.

No. 11.				
R	Tinct. ferri perchlor.	.	.	3j.
	Potassii bromidi	.	.	3j.
	Aquæ ad.	.	.	3j.

M.

No. 12.				
R	Ferri et ammoni citr.	.	.	3ss.
	Potassii iodidi.	.	.	3ss.
	Aquæ ad.	.	.	3j.

M.

They are all mixtures which have occurred in actual dispensing, the first being of a rather dangerous character.

The iron yields up its one and a half equivalent of chlorine in favour of one equivalent of iodine which it takes from one equivalent of iodide of potassium. There is thus half an equivalent of chlorine set free, which in turn combines with a fresh portion of potassium, setting free iodine; neither the iron nor the potassium having the power of combining with more than a single equivalent of iodine. Before the ingredients were mixed, there was neither free chlorine nor iodine, and none of them could be considered dangerous; after they are mixed iodine is set free, and danger is developed. The dark red brown colour and characteristic odour may be regarded as sufficient evidence of the free iodine, the presence of which may be made still more clear, by agitating a little of the mixture with sulphide of carbon, which takes the iodine from the aqueous solution, and acquires a beautiful violet colour, like the vapour of iodine.

A medical friend, who had been made aware of this decom-

position in the case of iodide of potassium, recently asked me if he would be safe in prescribing bromide of potassium in union with perchloride of iron. I replied in the affirmative, trusting to the fact that iron is capable of forming a sesquibromide, and the mixture was made similar to No. 11. The considerable increase in the depth of colour which took place on adding the bromide (which was added last), so far shook my confidence in my previously expressed opinion, that I thought it desirable to test the mixture with sulphide of carbon; but, as you see by the mixture I now make, there is no free bromine, or the sulphide would have become coloured yellow.

We also observe that the sulphide subsides to the bottom of the mixture No. 12 without becoming tinted, from which we learn that citrate of iron and ammonia affords us a suitable form for the administration of iron in connection with iodide of potassium.

Nos. 13, 14, and 15 exemplify the administration of iron salts with arsenic.

## No. 13.

R	Tr. ferri perchlor.	.	.	.	℥ss.
	Liq. arsenici hydrochlor.	.	.	.	gtt. xv.
	Aquæ	.	.	.	℥j.
M.					

## No. 14.

R	Tinct. ferri perchlor.	.	.	.	℥ss.
	Liq. arsenicalis	.	.	.	gtt. xv.
	Aquæ	.	.	.	℥j.
M.					

## No. 15.

R	Ferri et ammon. citr.	.	.	.	gr. v.
	Liq. arsenicalis	.	.	.	gtt. v.
	Aquæ	.	.	.	℥j.
M.					

The British Pharmacopœia arseniate of iron contains too little iron in a dose to be considered possessed of chalybeate properties; but any of the above forms may be considered satis-



factory, as they all retain the arsenic and iron permanently in solution.

Taking a general glance at the whole subject, and the illustrations I have given of it, there are one or two points to which I may again draw attention in conclusion.

That it is not desirable to go on multiplying without system and without principle the pharmaceutical preparations of iron.

That if it be desirable to give iron in states of combination in which it is not permanent, it would be more desirable to have formulas drawn up which would facilitate the extemporising of the required product rather than such formulas as are too complicated or tedious to be thus used, and so involve the making of an article which deteriorates by keeping; thus, if carbonate of iron be required, the "*Mist. Ferri Comp.*" is to be preferred to *Ferri Carbonas Saccharata*.

That the old, simple, well-tried, and permanent preparations of iron, if judiciously managed, are capable of meeting the requirements of very diverse circumstances.

That if the physician and the chemist consult together any difficulties of prescribing and dispensing are easily overcome; and that true progress in the pharmacy of iron will be best promoted by the physician pointing out what objections he finds in any of the established preparations, and indicating what desiderata are to be sought in the devising of new forms, and by his refusing to countenance preparations which are introduced without good evidence of their at least *seeking to attain* these objects; and the pharmacist's duty in this respect is to confine his attention to the chemistry and pharmacy of the subject in endeavouring to accomplish those objects which medical knowledge has shown to be desirable.

Such division of labour, and such harmonious working between the members of our calling and the members of the medical profession, cannot fail to be among the first-fruits of that higher pharmaceutical education which it has long been my habit to inculcate, and my desire to promote.

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